T. K. Chong

A Study of the Dehydration of some Typical Clays

A. STUDY OF THE DEHYDRATION OF SOME TYPICAL CLAYS

BY

TSENG KWAI CHOW
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THESIS

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I HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER MY

SUPERVISION BY Tseng Kwai Chow

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THE DEGREE OF MASTER OF SCIENCE

Cullen W. Parmelee
In Charge of Thesis

W. A. Noyes
Head of Department

Recommendation concurred in*

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____________________ Committee on Final Examination*
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*Required for doctor's degree but not for master's

A STUDY OF THE DEHYDRATION OF SOME TYPICAL CLAYS.

CONTENTS.

CHAPTER I.

INTRODUCTION.

One type of the hydrous silicates of alumina, which has the chemical formula $Al_2O_3.2SiO_2.2H_2O$, form a greater or smaller part of the pure and impure clays. Upon ignition they lose their combined water within a certain temperature range which is called the dehydration of clay. With this dissociation the character of the clays changes in an important manner. The property of plasticity is greatly diminished and in most cases is completely destroyed and deep-seated molecular transformations occur which are inferred by a decrease in specific gravity, increased solubility in acids and alkalies, and other decided alterations.

The dehydration temperatures of clays were not known with any degree of exactness until recently. An extensive research upon the dehydration of clays was carried on by G. H. Brown and E. T. Montgomery[1] who sought to determine the dehydration temperature range of a number of typical American clays, the loss of weight upon heating at constant temperaturs, the specific gravity after heating to several temperatures, and the loss of plasticity due to heating to several temperatures.

They determined the dehydration temperature range by comparing the heating curve of an electrically heated furnace with that obtained by measuring the temperature within a clay cylinder. The heating curves were plotted by the temperature against time.

The present study was undertaken for the purpose of discovering the dehydration temperatures of some typical clays. They are obtained by determining the dissociation points of the clays in corre-

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1. Technologic Papers of the Bureau of Standards No. 21.

lation with the vapor tension produced during decomposition by heating treatment and the dehydration curves were plotted by the vapor pressure against the corresponding temperature.

The suggestion for the apparatus was obtained from an article in Metallurgical and Chemical Engineering, Mar. 15, 1918, entitled "A Gas Dilatometer for ascertaining Decomposition point, Determination, of the Decomposition points of Cane Sugar, Dextrose, and Soluble Starch" by William C. Moore and B. Davies of the Research Laboratory of National Carbon Co., Inc., Cleveland, Ohio. A modification of the original apparatus was used for the determination of the dehydration curves of clays. The object of this investigation is to determine when the chemically combined water is given off; whether at one definite point- if so at what point, whether within some definite interval of temperature,- if so, at what interval; or whether the water is gradually driven off as the temperature increases.

CHAPTER II.

THE LITERATURE OF INVESTIGATIONS ON THE DEHYDRATION OF CLAYS.

For the dehydration temperatures of clay, Le Chatelier[1] assumed a series of ranges which depended upon the structure of the substance, varying from 150 to 770°C. Kaolin he supposed to dehydrate at 770°, this value being too high in the light of later measurements.

Ellis Lovejoy[2], an American investigator, determined the ignition loss of a No. 2 fire clay at the melting points of different metals and found the dissociation temperature to be near the fusion point of antimony, about 630°. As the result of similar work, A. E. Barnes[3], another American, reported the decomposition as taking place at about 565°. W. M. Kennedy[4], under the direction of Edward Orton, Jr., carried on a series of weighings of brickettes made from Forida kaolin, 30 grams in weight, at temperatures up to 1000°C. Here the loss of combined water was clearly shown to have taken place at 475°. The chemical study of dehydrated kaolin received attention by Hillebrand[5], F. W. Clarke[6], McNeil[7], Knote[8], and others.

I. Zs. physik, chemic, 1. p. 396; 1887 Comptes Rendu, 104, pp. 1443, 1517.

2. The clay Worker; 1888.

3. The clay Worker, 23, p. 705; June, 1895.

4. Trans. Am. Cer. Soc., 4. p. 152; 1902

5. U. S. Geol. Survey, Bull. 20, p. 98; 1885

6, Ibid., Bull. 125, p. 32.

7, J. Am. Chem. Soc., 28, p. 590.

8. Trans. Am. Cer. Soc. 12, p. 226; 1910.

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Knote[8], in a research on fire clays, showed by means of specifice gravity determinations that a sudden increase in volume occurs between 450° and 500°. The specific gravity reaches a miniumum at 550° in the case of a plastic clay and 600° with a Kentucky flint clay. For most clays, however, the point was found to be located at 550°. Beyond this temperature the density increased gradually up to 900°, when, between this temperature and 950°, a more or less rapid decrease in specific volume occurred. As regards the density drop at 550°, Knote suggests that the Al_2O_3 $2SiO_2$ $2H_2O$ breaks up to form Al_2O_3 SiO_2 + Al_2O_3 $3SiO_2$ + $4H_2O$, a reaction which is not very probable. The rise in density at 950°, accompanying an exothermic transformation, was noted by La Chatelier and by him ascribed to the polymerization of alumina.

The increase in volume coincident with dehydration has been noted by several earlier investigators. Ellis Lovejoy[9] noted that during this stage a decided expansion took place in bricks which he measured.

H. E. Ashley[10] summarized the facts known with reference to dehydration and reported some determinations of the dehydration range of a number of clays made in the clay products laboratory of the Bureau of Standards at Pittsburgh.

In heating clay an initial slow rate of heating was noted, due to the water held by the colloidal clay up to 300°. A retardation was then observed from 500° to 575°, due to the expulsion of the combined water. Following this stage a second retardation from

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[9] Trans. Am. Cer. Soc. 7.

[10] J. Ind. and Eng. Chem., 3. pp. 91-95; Feb., 1910.

about 825° to 925° and an acceleration occurring at about 925° were detected. The acceleration is the same which was previously found by Le Chatelier[11].

An investigation dealing with dehydration of clay has been reported by J. W. Mellor and A. D. Holdcroft[12]. The temperature range of the decomposition of kaolin was determined, and it was found that dehydration begins at no fixed temperature, but that the decomposition is appreciable at 500°. A marked lag appeared only between 600° and 700°. At about 900° the exothermic reaction was noted. The specific gravity of kaolin was reduced from 2.615 at 110° to 2.489 at 700°. Kaolin heated to 700° was found to be more soluble in hydrochloric acid than the untreated material.. The investigators conclude from the evidence obtained that upon dehydration the molecule breaks up into free silica, free alumina, and water. The exothermal change it is claimed, corresponds to a physical change of free alumina where-by the latter becomes less soluble in acids, less hygroscopic, and more dense. They say that a certain parallelism exists in the properties of alumina and calcined kaolin. On the basis of somewhat incomplete evidence Mellor and Holdcroft assume that the OH and Al groups are symmetrically placed in the molecule. Upon heating dehydrated kaolin under steam pressure these investigators succeeded in restoring 3 to 4 per cent water.

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[11]. See foot note 1.

[12]. Trans. Engl. Cer. Soc., 10. pp. 94-120.

An extensive research upon the dehydration of clays was carried on by R. Rieke[13], who sought to determine the differences in the dehydration temperatures of kaolins and clays, the possibility of definite dissociation temperatures and the loss of combined water at different temperatures. It was found by means of heating curves that heat absorption was observable at temperatures as low as 425° with some clays. The beginning of a pronounced halt, however, was never noted below 510° and for the average of 10 kaolins this point was about 538°. The lag extended to about 580° in most cases. Similar results were obtained with fire clays. The loss of weight increases rapidly with the temperature, since the velocity of the dissociation increases greatly as the temperature rises. Again, it was found that the decomposition was not completed at 700° after long continued heating, then remaining almost 0.5 percent to be driven off at a still higher temperature. Rieke found that plasticity was not necessarily destroyed upon dehydration. No evidence was produced which tended to show an unsymmetrical arrangement of the OH groups.

This question was likewise studied by A. M. Sokoloff[14] with special reference to the readiness with which the kaolin molecule is decomposed by a hot hydrochloric acid solution. Upon heating kaolin for five hours at different temperatures the following results were obtained.

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13. Sprechsaal, 44, pp. 637-641, 653-656.

14. Tonindustrie Zeitung, 36, 1107-1111.

Temperature °C	Loss in heating	Percent soluble al_2O_3
400	3.85 %	
500	11.70	28.99
600		33.74
700	12.94	33.96
800	13.00	34.67

The molecular ratio Al_2O_3 : H_2O for all temperatures approaches 1 : 2 quite closely.

G. H. Brown and E. T. Montgomery[15] compared a number of typical American clays as regards dehydration temperature range, loss of weight, specific gravity and loss of plasticity after heating, and summarized as follows.

The combined water is expelled at a comparatively low temperature so that even at 450°C the bulk of it may be driven off. In heating clay to constancy in weight at different temperatures the velocity of reaction is very slow at first and becomes suddenly rapid at 500°. The remaining portion of the water is removed more slowly, so that a temperature not less than 800° is required to accomplish complete expulsion.

The distinctness of the endothermal lag as well as the total loss in weight upon ignition (excluding abnormal loss due to carbon, sulphur, etc.) are criteria of the purity of clay. Like-wise the thermal acceleration at about 850° is shown more clearly by the higher types of clay.

A well defined minimum is shown in the specific gravity curve of clays, located at approximately 500°.

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[15] Bureau of Standards Tech. paper No. 21.

Dehydration does not necessarily destroy plasticity of clays and hence the combined water appears to have no direct connection with the phenomenon of plasticity. The surface clays examined lose their plasticity at the lowest temperatures, about 400°, followed by ball clay, at 450°, kaolin 500° to 600°, No. 2 fire clays 600° to 750° and shales 750° to 800°.

Ruby Wallach[16] heated clays slowly in an electric furnace from 20 to 1150°. A thermocouple in direct contact with the clay was connected to a galvanometer whose deflections were registered photographically. A sudden arrest in the heating curve for kaolin took place at 100° corresponding to the water held within the clay. A second break indicating the point where the dehydration proper begains, occurred at about 500°. The dehydration was invariably complete at 600°. A slow liberation of heat between 900° and 1000° was assumed to indicate the formation of an aluminate.

To detect the composition of the residue left after removing the H_2O from kaolin, Y. V. Samoilov[17] examined it by heating different varieties in an electric furnace connected with a self-registering pyrometer. From the curve representing the thermal changes, he obtained the following results. At 100-20° it loses hygroscopic H_2O, while its complete dehydration lies at 575°. Above 900° there is a sudden change indicating decomposition. Since no such change is observable when either Al_2O_3, SiO_2 or their mixture is heated to the same temperature, the composition of dehydrated kaolin cannot

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16. Compt. rend., 157, 48-50.

17. Bull. Acad. Sci., St. Petersburg, 1914, 779-94.

be Al_2O_3 + 2 SiO_2 as Al_2SiO_5 cannot be assumed to be changed by heat to $Al_2Si_2O_7$ (a substance unknown among minerals), the composition of calcined kaolin must be Al_2SiO_5 + SiO_2.

Ya. V. Samoilov[18] heated kaolin to high temperatures in 2 electric furnaces, in one of which the temperature rose faster than in the other. The kaolin curves showed 3 breaks, 2 corresponding to the endothermal effects of elimination of hygroscopic and of chemically bound water and 1 to the exothermal reaction of molecular rearrangement. The 1st 2 effects take place at about 565° on the slow, and at about 580° on the quick furnace. The molecular rearrangement on the slow furnace begins at about 900° - 910° and quickly reaches 990° while on the quick furnace it begins at about 935° and quickly reaches 1040°. Reduction of the speed of heating of the quick furnace, brought about by introducing additional resistance, lowered the temperature of the exothermal reaction. With a resistance of 9 ohms this temperature was the same as for the slow furnace, and with 14.3 ohms the reaction could not be observed at all.

J. W. Mellor[19] said there are three definite kaolinitic minerals corresponding with the empirical composition $Al_2O_3.2SiO_2.2H_2O$- kaolinite and nacrite are crystalline, the former is optically positive, and the latter optically negative, the third variety called clayite is colloidal. The distinction between halloysite and kaolinite or clayite can be readily demonstrated by time-temperature curves. Both halloysite and kaolinitic minerals show an exothermal critical point at about 900°, halloysite and colloidal clayite

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[18] Bull. Acad. Sci. Petrograd, 1915, 1759-68.

[19] Trans. Eng.Cer. Soc. 16, 73-84 (1917).

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each show an endothermal critical point at about 150°, and, unlike halloysite, kaolinite and clayite each shows one at about 500°. Clays (many ball clays and fire clays) with clayite as the dominant mineral show a terrace in the heating curve at about 150°, and this terrace is not shown by clays with kaolinite as the dominant mineral.

A. M. Sokolov[20] heated kaolinite from Gluchov from 1 to 5 hours at temperatures from 200 to 1000° and the loss of water was, 300° , 0.72; 400°, 0.67; 600°, 10.49; 700°, 11.92; 800°, 12.99. With more prolonged heating slightly higher results were obtained.

Shingo Satoh[21] a Japanese, investigated the effect of various temperatures upon Japanese crystalline kaolinite by means of both microscopic and thermal changes. He gave the thermal changes in 5 curves. (1) Heat absorption up to 100°, due to evaporation of occluded H_2O, (2) Weak heat evolution from 100° to 300°, due to oxidation of organic and other foreign materials; (3) Heat absorption from 450° to 700°, which might consist of 2 parts: (a) from 450° to 650°, due to dehydration; (b) from 650 to 700°, due to dissociation of anhydrous kaolinite to free alumina and silica; (4) heat evolution near 950°, due to polymerization of dissociated alumina and (5) heat evolution between 1200° and 1300°, due to formation of amorphous sillimanite. He suggests that thermal analysis might be used for grading different clays.

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20. Z. Kryst. Min. 55, 195-6; J. Chem. Soc. 112, II, 492; J. Soc. Chem. Ind. 36, 1177.

21. J. Chem. Ind. Japan 21, 631-48(1918)

CHAPTER III.

DESCRIPTION OF APPARATUS.

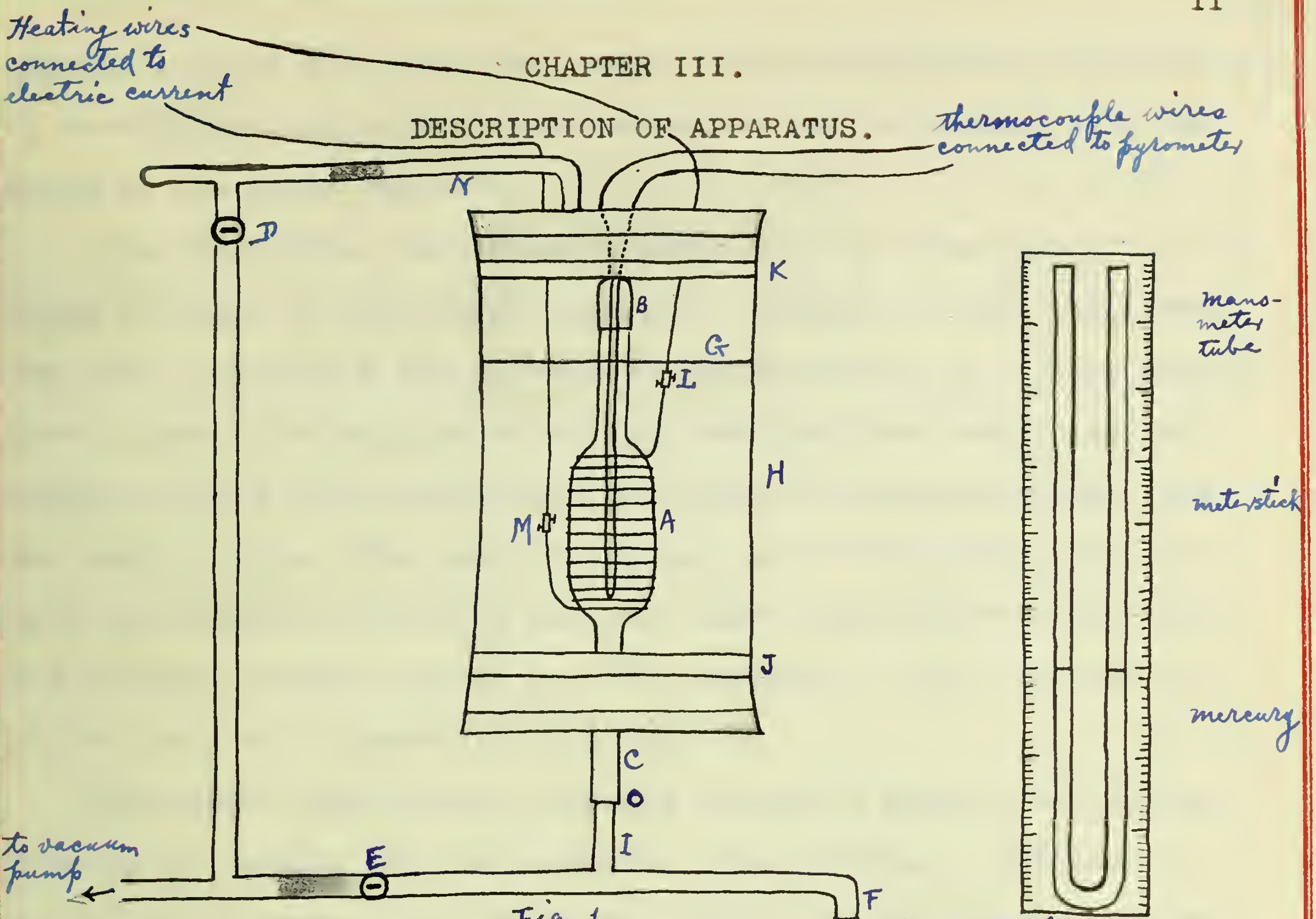

Fig 1.

The heating chamber A measures 90 mm. by 18 mm., and is drawn out at the lower end to join the tube I. The heating chamber A was wrapped with a thin sheet of mica over which nichrome wire was wound. At the outside of this was covered another thicker sheet of mica. The purpose of the inner sheet of mica was to prevent the glass from being heated too rapidly and that of outer sheet was to retain the heat within the chamber as much as possible. Mica disc G. and silocel discs J and K as shown in the figure were placed at the ends of the heating chamber for the same purpose of retaining the heat and preventing radiation..

A platinium-platinium-rhodium thermocouple, the platinium wire of which was insulated by a small porcelain tube which extended from the junction of the two wires to the end of tube B, was introduced into B so that the junction was in the center of the heating chamber

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and was covered with the clay sample. The thermocouple had previously been calibrated with a standard couple and was sealed into the glass at the upper end of B.

The whole tube, including A, B and part of C was placed in the glass cylinder H, with large hard cork stoppers at each end. Both the glass cylinder H and the whole heating tube A, B, C were made of Pyrex glass. The stopper at the top admitted the tube N and was pierced by the two thermocouple wires and two heating wires. The two heating wires were made of copper, one of which was connected with the nichrome wire at M and the other connected with the other end of the nichrome wire at L. The stopper at the lower end admitted the tube C penetrating through it.

The whole apparatus was clamped firmly in place- the cylinder H being horizontal and the manometer tube vertical. The shorter arm of the manometer tube measures 800 mm. and the longer, 900 mm. a meter stick was clamped behind the manometer tube in order that readings might be taken of the heights of the mercury columns. The manometer tube was placed in a constant temperature bath which was a box made of asbestos board (transite)½" thick. The inside dimensions of the box was 38" high, 9½" wide and 7" deep. At the front (38" by 9½") of the box had a glass window ½" from the right side and 1" from the bottom, to be 31½" high and 2" wide. This was to permit readings of the height of the mercury column to be taken from the outside. Hinges were put on the left side and a clasp on the right.

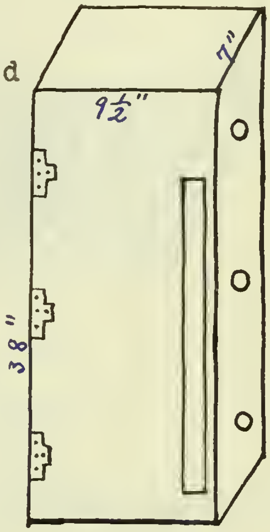

Fig 2

The box was made just large enough to hold the large ring-stand

which supported the manometer tube. The temperature of the bath had to be kept at 100°C. In the back of the box, at the top a hole was bored to allow the tube I (Fig. 1) to be put through. In the left side at the top a hole of the same diameter was made to receive the tube E F which connected with the vacuum pump. In the left side at the bottom, a hole ½" in diameter was bored to permit the entering of the wires for heating the box. Three more holes were made in the right side for putting in thermometers at the top, middle and bottom, the bulbs of the thermometers coming in just in front of the manometer tubes. The thermometers were put in cork stoppers to fit the holes.

To maintain the large box at a fairly constant temperature of 100°, two heaters were used, one at the bottom and the other at the center of the box. The heater used was of porcelain with coiled wire wound thru it and its size was 5½" by 6". The lead wires were brought in through the opening P made for them(Fig. 3).

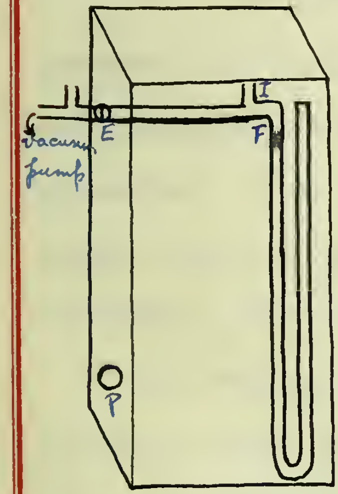

Fig 3

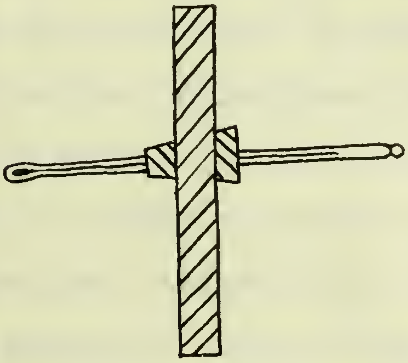

Fig 4

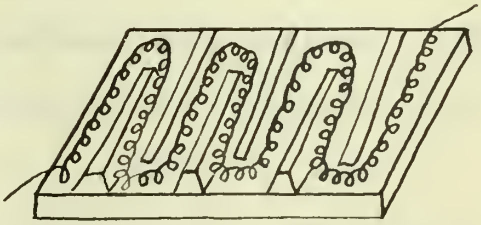

Fig 5

The arrangement of the apparatus outside the box was as follows; this being a view from above.

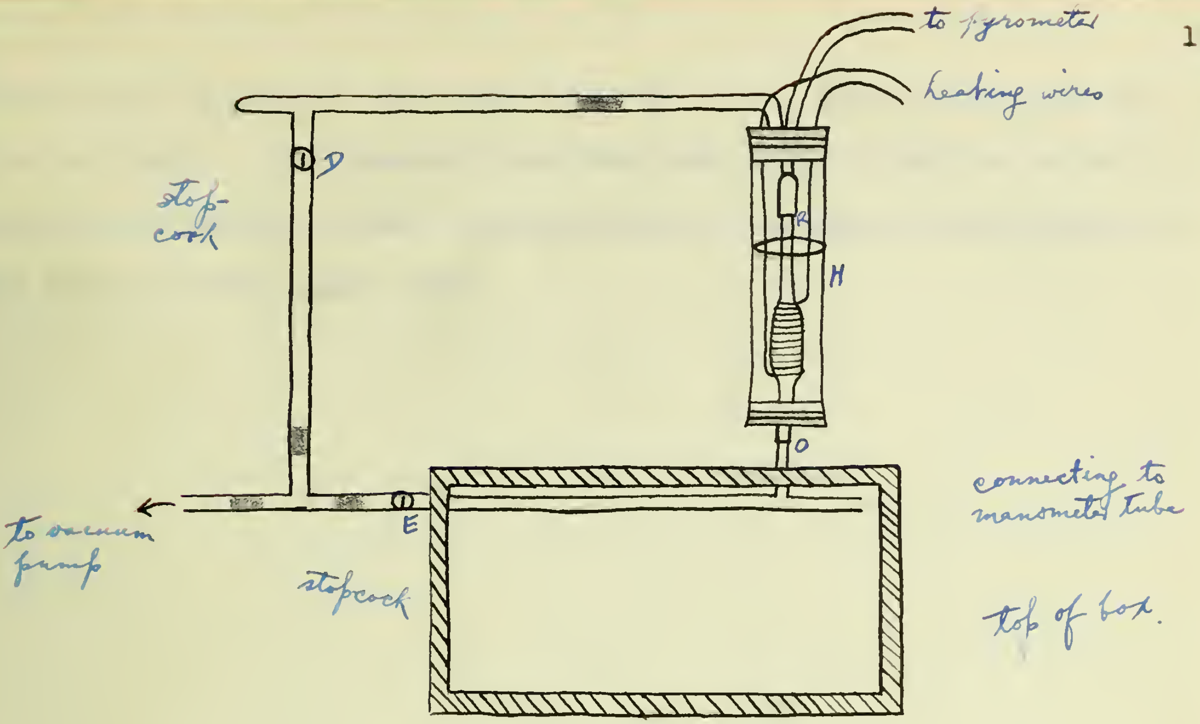

Fig 6.

It was necessary to have all heating wires and the tube connecting with the vacuum pump to lead away from the box in order to have the cylinder close to the box. A stopcock D was provided so that the vacuum in the cylinder could be sealed.

All connections were then firmly and securely sealed with beeswax and resin in order to make the apparatus air-tight. The thermocouple wires were connected by means of copper wires with a pyrometer, from which the temperature readings were derived. The copper heating wires were connected with an electric circuit, the strenght of the current being regulated by a resistance box.

The vacuum pump was started after enough mercury had been introduced into the manometer tube so that after evacuation was complete the difference in heights of the mercury in the two arms of the tube was equal to difference of heights of mercury in a barometer. In other words, the barometric pressure could be read directly from the manometer tube. When this state of evacuation was reached, the stop -cocks D and E (Fig. 6 and 1) were closed, thus

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sealing the vacuum in the whole system, i.e. both inner tube and outer cylinder. The vacuum pump was shut off. Then the electric current was turned on and the readings on manometer and pyrometer were taken at the same time.

CHAPTER IV.

THE TESTING OF APPARATUS.

Everything apparently being ready for use, it was yet not quite sure if the apparatus would work all right. So the heating chamber was filled with gypsum $CaSO_4.2H_2O$ which is known to contain 2 molecules of water which is given off at about 250°C. In this way the value and accuracy of the method might be tested.

Two grams of $CaSO_4.2H_2O$ were dried in an electric oven at 110°C, cooled over $CaCl_2$ and thoroughly mixed with 4 grams of $CaSO_4$ which had been dehydrated by heating to redness. After plugging the end of the furnace with glass wool, it was filled with the six grams of prepared gypsum, the thermocouple was introduced with the junction at the center of the mass, and the ends of the furnace were sealed with sealing wax.

The apparatus was then ready to be assembled. But it was difficult to make the whole apparatus air-tight. The cork stoppers at the two ends of the glass cylinder H (Fig. 1 and 6) were painted with hot wax to fill the pores and make them absolutely air-tight.

The apparatus was allowed to stand over night before being used. When the vacuum pump was started the next morning, some leaks were found especially at the joint O(Fig. 6) making it impossible to evacuate. After the leaks had been over-come by some means, the air bath was heated up to 100°C. Then the electric current was turned on and readings were taken.

Results: - Data. $CaSO_4.2H_2O$

Millivolts	Temp.C	Ht. of Hg.in high column	Ht. of Hg.in low column	Diff. in level of Hg.	Bar.Pres-diff. in level.
		840	100	740 mm	0
.78	117	832	108	724	16
.89	128	830	112	718	22
.90	139	820	121	699	41
1.00	150	814	126	688	52
1.10	163	814	127	687	53
1.15	172	814	127	687	53
1.20	182	815	126	689	51
1.30	194	818	122	696	44
1.40	207	822	118	704	36
1.65	235	825	115	710	30
1.90	260	826	115	711	29
2.05	273	827	114	713	27
2.15	283	828	113	715	25
2.33	300	829	113	716	24
2.41	310	828	114	714	26
2.50	318	826	115	711	29
2.60	328	826	115	711	29
2.70	338	823	118	705	35
2.82	353	820	122	698	42
2.92	363	815	127	688	52
3.00	373	810	132	678	62
3.20	390	802	138	664	76
3.28	400	796	145	651	89
3.32	405	793	148	645	95
3.39	413	790	152	638	102

Millivolts	Temp.C	Ht. of Hg. in high column	Ht. of Hg. in low column	Diff. in level of Hg.	Bar. Pres. diff. in level.
3.45	418	786	156	630	110
3.50	425	780	161	619	121
3.55	428	775	165	610	130
3.65	438	765	166	599	141
3.65	438	753	178	565	175
3.80	455	716	204	512	228
3.85	460	712	208	504	236
4.00	476	690	227	463	287
4.11	488	635	285	350	390
4.11	488	615	315	300	440
4.11	488	590	331	259	481
4.25	503	580	348	232	508
4.30	508	565	364	201	539
4.45	523	530	398	132	608
4.55	533	510	418	92	648
4.70	545	500	428	72	668
4.80	558	495	433	62	678
4.87	568	490	438	52	688
4.95	573	485	443	42	698
5.00	578	480	448	32	708
5.10	588	473	453	20	720

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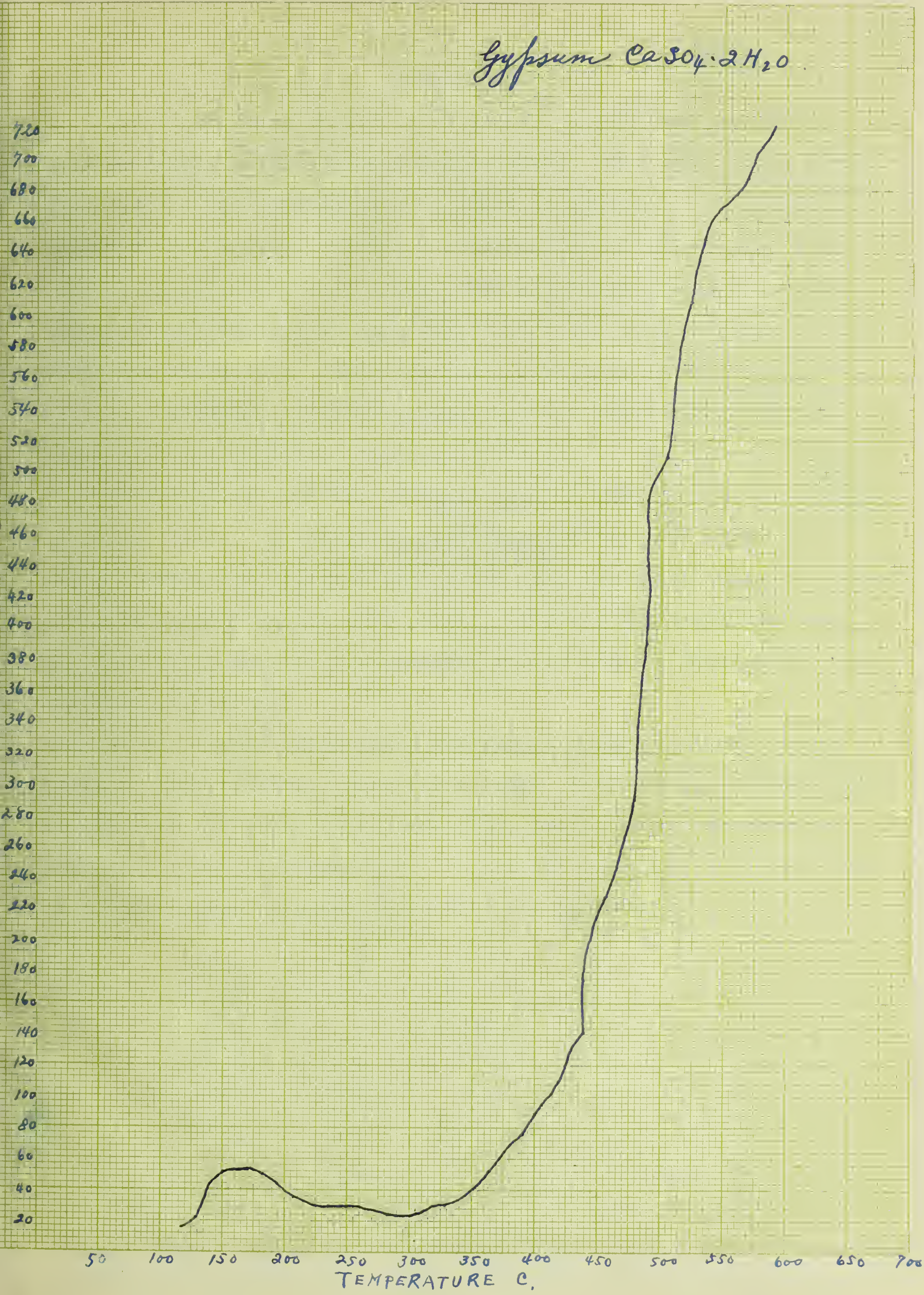
Gypsum $CaSO_4 \cdot 2H_2O$
720
700
680
660
640
620
600
580
560
540
520
500
480
460
440
420
400
380
360
340
320
300
280
260
240
220
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TEMPERATURE C.

Conclusion.

The first break in the curve occurs between 125° and 150°, this evidently being the hygroscopic water. The drop in the curve from 175° to 300° indicates a decrease in pressure. This might be caused by two things. First, a condensation of water which was just given off might take place. But all tubes containing water vapor were maintained at approximate 100°, so that condensation of water was impossible. No evidences of droplets of water could be noticed. Secondly, it is possible that the water given off combined with the dehydrated calcium sulphate.

From 300° to 440° the curve is regular showing that a change is taking place. Quite uniformly, from 440° to 560° there is a rapid increase in pressure indicating that the water vapor was given off more rapidly during this period. The run was continued until the difference between the pressure within and the atmospheric pressure was only 43 mm.

It was thought when there was such a sudden increase in the pressure that possibly there was a leak. But when the furnace was cooled, and the water vapor condensed, the pressure decreased almost to the original pressure. So all the increase in pressure must have been caused by gases given off from the $CaSO_4$.

CHAPTER V.

THE TESTING OF APPARATUS (CONTINUED).

Although the apparatus had been tested by using gypsum $CaSO_4 \cdot 2H_2O$, which would dehydrate at a definite temperature, yet it was still safer to test the apparatus again by using some other substances which possess no chemically combined water. For this purpose, the pure sand and the fused alumina were taken as the testing materials.

I first took 100 grams of sand and heated it to red heat in a furnace for two hours. Then placed in a desiccator over $CaCl_2$ and let it cool down. Then I quickly weighed out 5 grams and put in the apparatus.

The apparatus was then assembled and any leakage was eliminated. The sand was heated in the furnace as usual to above 500°C. The changes in manometer and pyrometer readings were noted and the results are given in the following data.

Millivolts	Corrected millivolts	Temp.C	Hg.height in high col.	Hg.height in low col.	Diff.in level of Hg. col.	Bar.P-diff.in level.
			85.0	11	74	0
0.50	.62	95	84.9	11.1	73.8	2
0.99	1.11	165	84.8	11.2	73.6	4
1.39	1.51	224	84.7	11.3	73.4	6
1.60	1.72	240	84.5	11.5	73.0	10
1.80	1.92	260	84.4	11.6	72.8	12
2.00	2.12	280	84.3	11.7	72.6	14
2.13	2.25	290	84.2	11.8	72.4	16
2.21	2.33	300	84.1	11.9	72.2	18
2.30	2.42	310	84.0	12.0	72.0	20
2.40	2.52	320	83.8	12.2	71.6	24

Millivolts	Corrected millivolts	Temp.C	Hg.height in high col.	Hg. height in low col.	Diff.in level of Hg. col.	Bar.P-diff.in level
2.55	2.67	335	83.6	12.4	71.2	28
2.65	2.77	345	83.5	12.5	71.0	30
2.83	2.95	365	83.4	12.6	70.8	32
2.95	3.07	378	83.3	12.7	70.6	34
3.00	3.12	385	83.2	12.8	70.4	36
3.15	3.27	400	83.1	12.9	70.2	38
3.30	3.42	415	82.9	13.1	69.8	42
3.40	3.52	425	82.7	13.3	69.4	46
3.50	3.62	435	82.6	13.4	69.2	48
3.60	3.72	445	82.5	13.5	69.0	50
3.70	3.82	458	82.4	13.5	68.9	51
3.80	3.92	470	82.4	13.6	68.8	52
3.90	4.02	480	82.3	13.6	68.7	53
4.00	4.12	490	82.3	13.7	68.6	54
4.10	4.22	500	82.2	13.7	68.5	55
4.29	4.41	517	82.2	13.8	68.4	56

In the next test, the fused alumina called alundum was used. Took five grams of alumdum and heated it in a porcelain crucible over a blast lamp to redness for about two hours. Cooled in a desiccator. The sample was then transferredin the furnace.

The apparatus was reassembled as before and the vacuum pump was started to evacuate the whole apparatus. The electric current was turned on and the furnace containing alundum was heated gradually to above 500°C. The changes of pressure and temperature on the manometer and the pyrometer respectively were watched carefully and their readings were recorded simultaneously. The results are given in the following data and the curve plotted was shown in the

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accompanying diagram.

Millivolts	Corrected Millivolts	Temp.C room-temp. 24°C.	Hg.height in high column.	Hg ht.in low col	Diff,in level of Hg.col.	Bar.P-diff. in leve
			84.6	9.6	75.0	0
.15	27	43	84.6	10.6	74.0	10
.65	77	118	84.6	10.6	74.0	10
1.01	113	165	84.6	10.6	74.0	10
1.38	150	215	84.6	10.6	74.0	10
1.95	207	273	84.6	10.6	74.0	10
2.00	212	278	84.6	10.6	74.0	10
2.29	241	310	84.6	10.6	74.0	10
2.50	262	330	84.6	10.8	73.8	12
2.64	276	345	84.6	10.8	73.8	12
2.70	282	352	84.6	10.9	73.7	13
2.72	284	354	84.5	11.0	73.5	15
2.80	292	362	84.5	11.1	73.4	16
2.90	302	372	84.5	11.2	73.3	17
3.00	312	385	84.4	11.3	73.1	19
3.16	328	400	84.3	11.4	72.9	21
3.20	332	407	84.3	11.5	72.8	22
3.35	347	420	84.3	11.5	72.8	22
3.44	356	428	84.3	11.5	72.8	22
3.56	368	440	84.2	11.6	72.6	24
3.66	378	452	84.2	11.6	72.6	24
3.72	384	458	84.1	11.7	72.4	26
3.88	400	475	83.9	11.9	72.0	30
3.93	405	481	83.7	12.0	71.7	33
4.05	417	493	83.7	12.0	71.7	33
4.12	424	500	83.5	12.1	71.4	36
4.30	442	520	83.4	12.2	71.2	38

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From the results obtained in the last test we plotted a curve NO3 which shows there was some hygroscopic water present, although the alundum had been calcined at a strong red heat for more than 2 hours. So this time we tried the same substance, i. e. alundum, changing the running of the experiment slightly. The procedure may be briefly stated as follows:

Weighed out about five grams of alundum in a porcelain crucible and heated it strongly over a blast lamp for nearly two hours. Then took it off and placed it in a desiccator over $CaCl_2$ to let it cool. The sample was then introduced into the furnace and the whole apparatus was reassembled as usual.

After the apparatus was evacuated, the sample in the furnace was heated up to 150°C and then the furnace was evacuated again. As soon as it was evacuated, the temperature was raised and it was watched carefully to see whether there was any more water vapor left in the apparatus. It was seen that when the temperature rose the pressure increased slowly so that the mercury height in high column dropped little by little. The latter fact indicates that a small amount of water was still present. So the apparatus was evacuated once more to remove all the water vapor present.

Then the temperature was raised again above 500°C. The temperature and pressure changes were read from the pyrometer and manometer respectively at very short intervals. The results are stated in the following data and the curve is plotted in the accompanying diagram.

Millivolts	Corrected millivolts	Temp.C room T.	Hg.height in HighCol.	Hg.height in lowCol.	Diff.in level of Hg.Col.	Bar.P.-Diff.in level.
		21.5°C	842	104	738	0
45	57	80	850	105	745	-7
90	102	150	850	105	745	-7
146	158	225	850	105	745	-7
180	192	260	850	105	745	-7
196	208	272	850	105	745	-7
210	222	288	850	105	745	-7
226	238	305	850	105	745	-7
241	253	322	850	105	745	-7
270	282	352	850	105	745	-7
303	315	388	851	104	747	-9
320	332	405	851	104	747	-9
355	367	442	851	104	747	-9
380	392	458	851	104	747	-9
400	412	488	851	104	747	-9
410	422	499	851	104	747	-9
430	442	519	851	104	747	-9
438	450	525	851	104	747	-9

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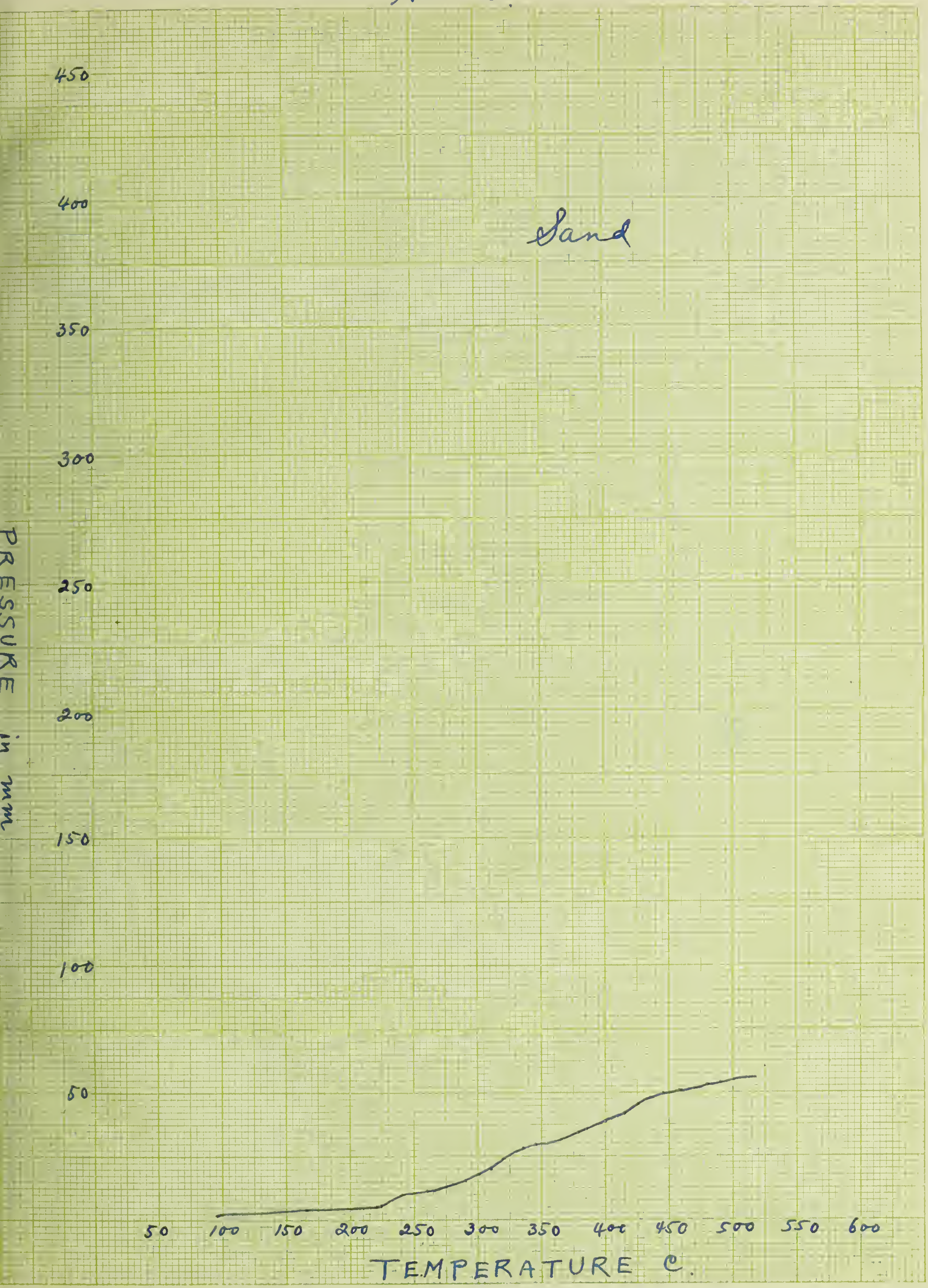
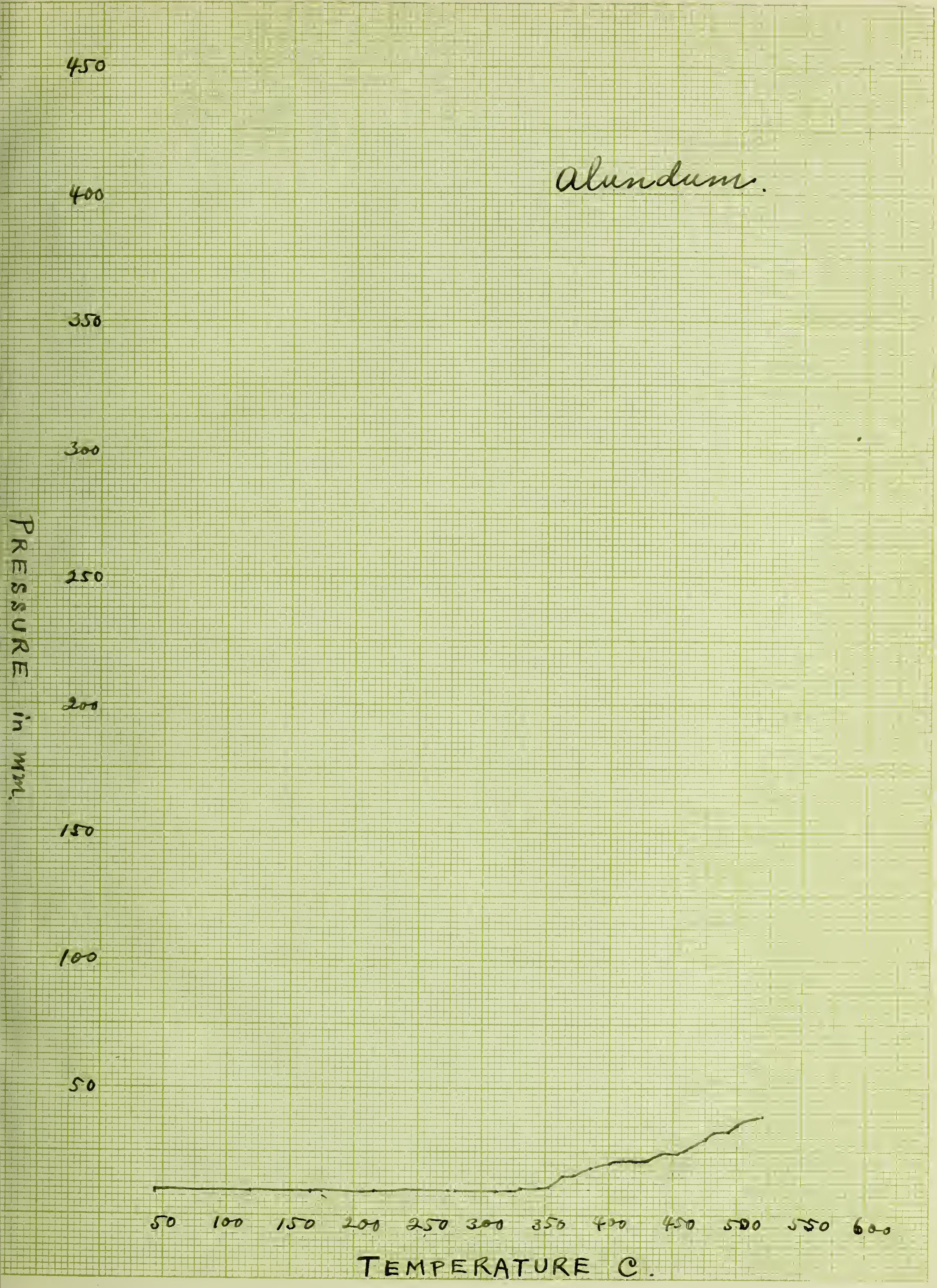
No. 2.


Sand
PRESSURE in mm
450
400
350
300
250
200
150
100
50
50
100
150
200
250
300
350
400
450
500
550
600
TEMPERATURE C.

No. 3


Alundum.
PRESSURE in MM.
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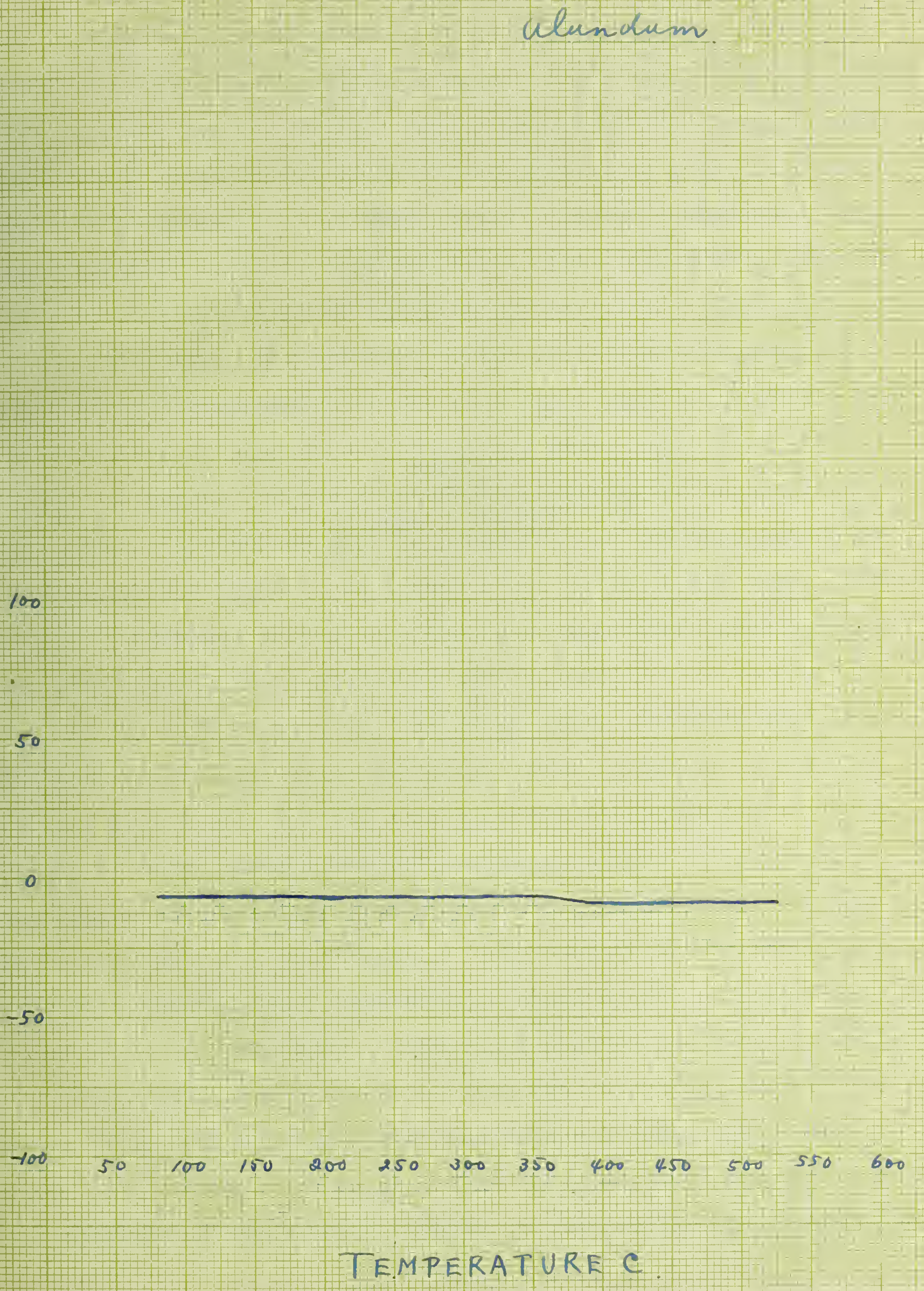
TEMPERATURE C.

No. 4


alundum
100
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PRESSURE in mm.
50
100
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550
600
TEMPERATURE C

Conclusion.

In the case of sand the curve goes up quite smoothly which shows there was some hygroscopic water in the sand, although it had been calcined for two hours. But by observing the curve we know that only a small amount of water was present so that the vapor pressure produced by it is very little. As the temperature rose, the volumes of furnace, sand and water vapor increases at the same time, but the expansion of the last one was much greater than either of the other two. The expanded volume of the furnace was just balanced by the expansion of the sand and a part of the water vapor The expansion of some other part of the water vapor still left caused the mercury in the high column of manometer to drop. This fact means a slight increase of pressure. As the temperature became higher and higher, the pressure due to the water vapor produced also increased proportionally, i. e. the water vapor expanded more and more as the temperature was raised.

In the case of alundum, the first curve indicates that there was some hygroscopic water contained in it, although it was previously heated over a blast lamp strongly for some time. The amount of water vapor given off, however, seemed to be a small quantity which was shown by the pressure produced by the vapor. Since the apparatus was evacuated only once at the beginning before being heated, the presence of a small amount of hygroscopic water in it is reasonable. The first part of the curve is quite flat, because during that period both the apparatus, alundum and water vapor were expanding and just balanced each other. The latter part of the curve denotes that the pressure increased with the increase of temperature because at that period the apparatus

somewhat reached its maximum expansion point while the water vapor was still expanding so that it caused the increase of pressure.

The second curve is quite flat through-out and shows that there is no moisture in the furnace; for it was evacuated first and then heated up to 150°C and then evacuated again. This process was repeated once more until all the water vapor present was removed by the vacuum pump. When the temperature rose gradually, the volume of furnace expanded with the temperature. Consequently, the mercury in the high column came up and the pressure decreased and gave a negative value.

CHAPTER VI.

Determination of dehydration temperature range of Tennessee Ball clay No. 3.

In the first determination, Tennessee Ball clay No. 3 from Mandle Clay Co., was used, the sample being prepared in the following way. Two grams of Tennessee Ball clay were dried in an electric oven at 110°C, cooled over $CaCl_2$ in a desiccator and thoroughly mixed with four grams of Tennessee Ball clay which had been dehydrated by heating to redness. After plugging the end of the furnace with glass wool, it was filled with the six grams of prepared mixture. The thermocouple was then introduced into the furnace with the junction at the center of the mass, and the end R (Fig. 6) of the furnace was sealed with sealing wax. The apparatus was then ready to be assembled. But some leak was found at the end O (Fig. 6) and the latter could not be made air tight by merely painting with the mixture of paraffine and resin. Finally it was wrapped with hard rubber tubing around it by some iron wire and the leakage was over come.

Then the apparatus was let stand for few hours before being used. When the vacuum pump was started and the whole system evacuated, the manometer box was heated to 100°C. Then the electric current was turned on and the readings of temperature on this pyrometer and of the pressure on the manometer were taken at intervals of one minure. Before the run, the barometer pressure was first recorded. The results obtained are stated in the following data.

Millivolts	Corrected millivolts	Temp.C	Ht.of Hg in h.Col.	Ht.ofHg in l.Col.	Diff.in level of Hg.Col.	Bar. P.- diff. in level.
		room temp.	85.0	11.1	73.9	0
1.00	112	165	82.5	13.6	68.9	50
1.21	133	195	82.4	13.8	68.6	53
1.45	157	225	82.3	13.9	68.4	55
1.65	177	245	82.2	14.0	68.2	57
1.79	191	260	82.1	14.0	68.1	58
1.91	203	268	82.0	14.0	67.9	60
2.00	212	277	82.0	14.1	67.9	60
2.10	222	288	82.1	14.1	68.0	59
2.20	232	300	82.1	14.2	67.9	60
2.30	242	310	82.1	14.2	67.9	60
2.31	243	311	82.0	14.3	67.7	62
2.34	246	314	81.8	14.5	67.3	66
2.41	253	320	81.5	14.8	66.7	72
2.45	257	325	81.3	15.0	66.3	76
2.55	267	335	81.0	15.3	65.7	82
2.59	271	340	80.8	15.5	65.3	86
2.60	272	342	80.5	15.8	64.7	92
2.60	272	342	80.4	15.9	64.5	94
2.60	272	342	80.2	16.0	64.2	97
2.60	272	342	80.0	16.3	63.7	102
2.61	273	344	79.5	16.6	62.9	110
2.70	282	350	79.0	17.2	61.8	121
2.77	289	360	78.7	17.5	61.2	127
2.80	292	363	78.2	18.0	60.2	137
2.85	297	370	77.9	18.3	59.6	143

Millivolts	Corrected millivolts	Temp.C.	Ht.of Hg in h.Col.	Ht.ofHg in l.Col.	Diff.in level of Hg.Col.	Bar. P. diff. in level.
2.90	302	373	77.5	18.6	58.9	150
2.91	303	375	77.1	19.0	58.1	158
2.95	307	378	76.9	19.2	57.7	162
3.00	312	384	76.3	19.7	56.6	173
3.05	317	389	75.5	24.0	51.5	224
3.10	322	394	75.2	20.7	54.5	194
3.10	322	394	75.0	20.9	54.1	198
3.13	325	398	74.9	21.0	53.9	200
3.15	327	400	74.9	21.0	53.9	200
3.15	327	400	74.9	21.0	53.9	200
3.15	327	400	75.0	21.0	54.0	199
3.20	332	405	74.4	21.5	52.9	210
3.28	340	413	73.7	22.2	51.5	224
3.40	352	425	73.3	22.6	50.7	232
3.45	357	430	73.2	22.7	50.5	234
3.51	363	435	73.2	22.7	50.5	234
3.52	364	436	72.9	23.0	49.9	240
3.64	376	450	72.4	23.5	48.9	250
3.70	382	457	72.1	23.8	48.3	256
3.70	382	457	71.8	24.1	47.7	262
3.70	382	457	71.6	24.3	47.3	266
3.71	383	458	71.4	24.5	46.9	270
3.70	382	457	71.0	24.9	46.1	278
3.70	382	457	70.7	25.0	45.7	282
3.70	382	457	70.5	25.2	45.3	286
3.65	377	450	70.2	25.5	44.7	292

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Millivolts	Corrected Millivolts	Temp.C.	Ht.of Hg in h.Col.	Ht.ofHg in l.Col.	Diff.in level of Hg.Col.	Bar. P.- diff. in level.
3.61	373	446	70.0	25.7	44.3	296
3.61	373	446	69.8	25.9	43.9	300
3.61	373	446	69.4	26.3	43.1	308
3.63	375	450	69.2	26.5	42.7	312
3.65	377	453	69.1	26.6	42.5	314
3.70	382	458	68.7	26.9	41.8	321
3.73	385	460	68.4	27.2	41.2	327
3.80	392	468	68.2	27.4	40.8	331
3.83	395	470	68.0	27.5	40.5	334
3.80	392	468	67.9	27.7	40.2	337
3.88	400	475	67.5	27.9	39.6	343
3.95	407	483	67.2	28.2	39.0	349
4.03	415	490	66.7	28.8	37.9	360
4.03	415	490	66.4	29.1	37.3	366
4.06	418	495	66.1	29.3	36.8	371
4.06	418	495	65.9	29.5	36.4	375
4.10	422	498	65.8	29.7	36.1	378
4.10	422	498	65.6	29.8	35.8	381
4.10	422	498	65.4	30.0	35.4	385
4,10	422	498	65.3	30.1	35,2	387
4.12	424	500	65.2	30.2	35.0	389
4.15	427	504	65.0	30.4	34.6	393
4.15	427	504	64.8	30.6	34.2	397
4.15	427	504	64.6	30.8	33.8	401
4.15	427	504	64.4	30.9	33.5	404
4.13	425	502	64.3	31.1	33.2	407

Millivolts	Corrected Millivolts	Temp.C.	Ht.of Hg in H.Col.	Ht. of Hg in L. Col.	Diff.in level of Hg.Col.	Bar. P.— diff. in level.
4.13	425	502	44.2	31.2	33.0	409
4.13	425	502	64.0	31.3	32.7	412
4.13	425	502	63.9	31.4	32.5	414
4.13	425	502	63.9	31.5	32.4	415
4.13	425	502	63.8	31.6	32.2	417
4.20	432	510	63.8	31.6	32.2	417
4.20	432	510	63.7	31.6	32.1	418
4.20	432	510	63.6	31.7	31.9	420
4.20	432	510	63.5	31.8	31.7	422
4.20	432	510	63.4	32.0	31.4	425
4.20	432	510	63.3	32.1	31.2	427
4.20	432	510	63.0	32.2	30.8	431
4.20	432	510	62.8	32.5	30.3	436
4.20	432	510	62.6	32.6	30.0	439
4.20	432	510	62.5	32.7	29.8	441
4.20	432	510	62.1	33.1	29.0	449
4.20	432	510	61.8	33.4	28.4	455
4.20	432	510	61.7	33.5	28.2	457
4.29	441	517	61.3	33.9	27.4	465
4.38	450	525	60.9	34.3	26.6	473
4.38	450	525	60.6	34.6	26.0	479
4.43	455	530	60.3	34.9	25.4	485
4.45	457	535	60.0	35.1	24.9	490
4.45	457	535	59.5	35.6	23.9	500
4.45	457	535	58.5	36.5	22.0	519
4.45	457	535	58.3	36.8	21.5	524

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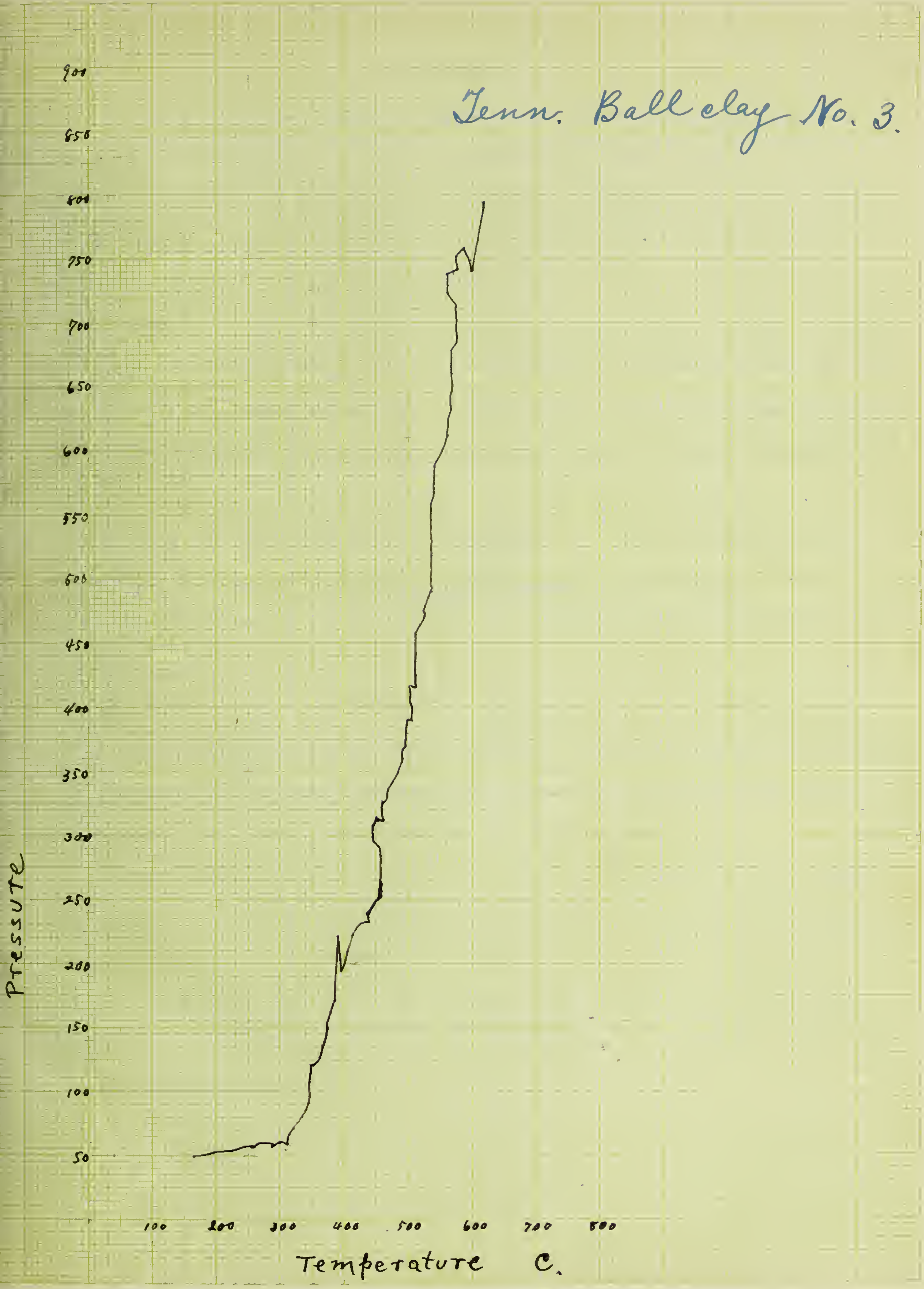
Millivolts	Corrected millivolts	Temp.C.	Ht. of Hg in H.Col.	Ht.of Hg in L.Col.	Diff.in level of Hg.Col.	Bar.P.— diff. in level.
4.45	457	535	57.9	37.1	20.8	531
4.45	457	535	57.6	37.5	20.1	538
4.45	457	535	57.0	38.1	18.9	550
4.45	457	535	56.5	38.5	18.0	559
4.49	461	540	56.1	38.8	17.3	566
4.49	461	540	55.8	39.0	16.8	571
4.49	461	540	55.5	39.3	16.2	577
4.49	461	540	55.0	39.8	15.2	587
4.71	483	560	53.7	40.9	12.8	611
4.74	486	565	52.6	41.9	10.7	632
4.74	486	565	52.2	42.4	9.8	641
4.79	491	569	51.7	42.9	8.8	651
4.79	491	569	51.3	43.2	8.1	658
4.79	491	569	51.0	43.6	7.4	665
4.79	491	569	50.5	43.9	6.6	673
4.79	491	569	50.3	44.2	6.1	678
4.83	495	575	50.0	44.5	5.5	684
4.83	495	575	49.7	44.8	4.9	690
4.83	495	575	49.1	45.3	3.8	701
4.83	495	575	48.5	45.9	2.6	713
4.71	483	560	48.0	46.4	1.6	723
4.71	483	560	47.7	46.7	1.0	729
4.71	483	560	47.4	46.9	5	734
4.71	483	560	47.2	47.1	1	738
4.80	492	569	47.1	47.2	-1	740
4.85	497	575	47.0	47.3	-3	742

Milli volts	Corrected Millivolts	Temp.C.	Ht.of Hg in H.Col.	Ht.of Hg in L.Col.	Diff.in level of Hg.Col.	Bar.P.— diff. in level.
4.85	497	575	46.5	47.8	-13	752
5.00	512	588	46.2	48.2	-20	754
5.09	521	598	46.5	47.5	-10	749
5.10	522	599	47.1	47.3	-2	741
5.10	522	599	47.2	47.3	-1	740
5.13	525	600	47.2	47.3	-1	740
5.28	540	615	44.5	50.0	-55	794

No. 5


Tenn. Ball clay No. 3.
900
850
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750
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100
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Pressure
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700
800
Temperature C.

Conclusion.

In this case the vapor pressure increases quite rapidly as the temperature rises. Since the Tennessee ball clay No. 3 is an impure clay, the pressure produced by dehydration of clay does not represent the true pressure of combined water in clay owing to the presence of some organic matter.

As is to be anticipated, the impure clay shows a more marked increase in pressure, i. e. the increase of pressure due to the freeing of the combined water in clay, than the purer ones, because the vapor pressure is somewhat interfered with by that produced by the decomposition of the organic substance in the clay.

It may be observed on the curve No. 5 that the dehydration seems to begin at about 310°C. Below this temperature all the water vapor produced is of course, the mechanically held water in the clay.

No water vapor was seen to be condensed in the heating chamber. The determination was made in about three hours and the electric current during that period was increased gradually by regulating the resistance box inserted in the circuit.

Some breaks occurin the curve. The manometer tube was carefully watched during the determination, and at these points there was an increase in pressure followed by a decrease , probably due to cooling of parts of the system, the temperature remaining constant. At 560° the pressure of the water vapor became equal to atmospheric pressure.

CHAPTER VII.

DETERMINATION OF DEHYDRATION TEMPERATURE RANGE OF GEORGIA KAOLIN.

In the determination of dehydration temperature of Tennessee ball clay No. 3 (i.e. last experiment), the heating chamber which was made of Pyrex glass was heated up to 615°C. This temperature seems to be too high for Pyrex glass to withstand, so that the heating chamber was collapsed and broken. Consequently, a new Pyrex glass tube was made which had the same size as the old one, the only difference being that the ground joint O (Fig. 6) was omitted and changed to ordinary glass tubing shape.

In this experiment Georgia Kaolin was used. A mixture of one gram of Georgia Kaolin and four grams of the same previously dehydrated by heating to redness for two -three hours was taken which was prepared in the same way as before. The prepared sample was then filled into the furnace. This time the writer did not use the glass wool to plug the ends of the furnace, because he found that some of the glass wool was fused and formed a hard mass in the last determination which was inconvenient to be removed from the furnace.

The apparatus was reassembled and the whole system was tested for leakage. As soon as everything was all right, the furnace was heated up gradually by turning on the electric current and the readings of temperature were recorded at about every one or two minutes. The results, obtained are given in the following data.

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Millivolts	Corrected Millivolts	Temp.C.	Hg Ht. in H. Col.	Hg Ht. in L. Col.	Diff. in level of Hg Col.	Bar. P.— diff. in level.
		room temp. 23°C	84.3	10.2	74.1	0
41	53	75	83.9	10.6	73.3	8
86	98	145	83.1	12.2	70.9	32
169	181	250	82.9	12.5	70.4	37
202	214	280	82.8	12.6	70.2	39
226	238	305	82.7	12.7	70.0	41
242	254	323	82.6	12.8	69.8	43
254	266	335	82.5	12.9	69.6	45
260	272	342	82.4	12.9	69.5	46
265	277	345	82.3	13.0	69.3	48
270	282	352	82.3	13.1	69.2	49
275	287	357	82.2	13.2	69.0	51
281	293	362	82.1	13.3	68.8	53
281	293	362	82.0	13.4	68.6	55
290	302	373	81.8	13.6	68.2	59
295	307	377	81.7	13.7	68.0	61
300	312	384	81.6	13.9	67.7	64
300	312	384	81.5	14.0	67.5	66
318	330	404	81.2	14.4	66.8	73
325	337	410	81.0	14.6	66.4	77
330	342	415	80.8	14.7	66.1	80
334	346	418	80.7	14.8	65.9	82
339	351	425	80.5	15.0	65.5	86
345	357	430	80.1	15.4	64.7	94
350	362	437	80.0	15.5	64.5	96
353	365	440	79.9	15.6	64.3	98

Millivolts	Corrected Millivolts	Temp.C.	Hg Ht. in H. Col.	Hg Ht.in L. Col.	Diff.in level of Hg Cols.	Bar. P.-diff. in level.
355	367	443	79.7	15.8	63.9	102
360	372	448	79.6	15.9	63.7	104
360	372	448	79.5	16.0	63.5	106
360	372	448	79.3	16.1	63.2	109
360	372	448	79.2	16.2	63.0	111
360	372	448	79.1	16.3	62.8	113
360	372	448	79.0	16.4	62.6	115
360	372	448	78.9	16.5	62.4	117
360	372	448	78.8	16.6	62.2	119
360	372	448	78.7	16.7	62.0	121
360	372	448	78.6	16.8	61.8	123
360	372	448	78.5	16.9	61.6	125
360	372	448	78.4	17.0	61.4	127
360	372	448	78.3	17.1	61.2	129
360	372	448	78.1	17.3	60.8	133
365	377	453	78.0	17.4	60.6	135
365	377	453	77.9	17.4	60.5	136
365	377	453	77.7	17.6	60.1	140
378	390	465	76.9	18.3	58.6	155
387	399	475	76.3	18.9	57.4	167
387	399	475	75.8	19.3	56.5	176
391	403	478	75.5	19.6	55.9	182
391	403	478	75.3	19.8	55.5	186
395	407	483	75.1	20.0	55.1	190
395	407	483	74.9	20.2	54.7	194
400	412	488	74.5	20.5	54.0	201

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Millivolts	Corrected Millivolts	Temp.C.	Hg Ht.in H.Col.	Hg Ht.in L.Col.	Diff.in level of Hg.Cols.	Bar. P.-diff. in level.
400	412	488	74.4	20.6	53.8	203
402	414	490	74.1	21.0	53.1	210
402	414	490	73.4	21.0	52.0	221
420	432	508	72.5	22.4	50.1	240
426	438	514	71.9	23.0	48.9	252
427	439	515	71.6	23.3	48.3	258
430	442	518	71.5	23.4	48.1	260

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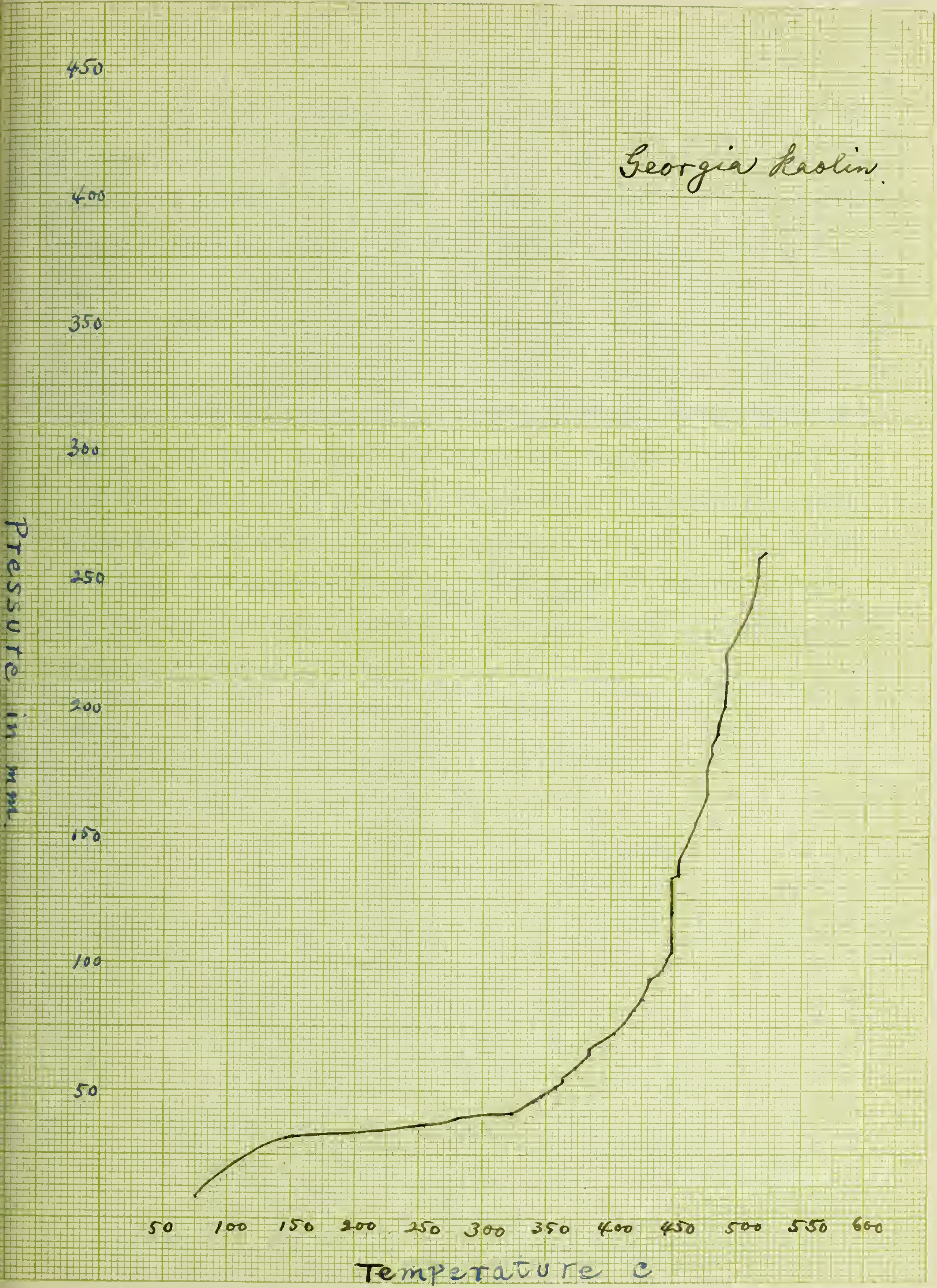
No. 6


Georgia Kaolin.
Pressure in mm.
450
400
350
300
250
200
150
100
50
50
100
150
200
250
300
350
400
450
500
550
600
Temperature C

Conclusion.

As previously stated, the Pyrex glass furnace could not withstand the temperature as high as 615°C under reduced pressure and it generally will collapse when heated to about 530°C under atmospheric pressure, so in this determination the temperature was raised only to about 520°C in order to save the glass furnace.

By inspecting the curve No. 6 we can tell the physically combined water was given chiefly off from 75° to 145°C. From 145° to 323° the curve is nearly flat, showing that not much change took place. From 323° to 448° more water was evolved. From 448° to 490° there is a rapid increase in pressure, the chemically combined water was given off beginning at about 325°. This period is called the dehydration temperature range of Georgia Kaolin. The run was continued until the temperature reached 518°C and the pressure continued to rise.

CHAPTER VIII.

DETERMINATION OF DEHYDRATION TEMPERATURE RANGE OF NORTH CAROLINA KAOLIN.

The apparatus was reassembled as before and the heating chamber was filled with a sample of Harris North Carolina kaolin from Harris Kaolin Co., Spruce Pine, North Carolina, which was prepared in a similar way as previously described in other experiments. This sample consisted of one gram of raw clay and four grams of dehydrated.

The barometric pressure reading just before the time of running was noted and the vacuum pump was started. The whole apparatus was carefully examined to see if there might be any leaks in it. It was found that the manometer reading inside the manometer box gave the same number of millimeters of pressure as that indicated by barometer. This fact shows there was no leakage in the whole system.

Then the vacuum pump was shut off, and the manometer box was heated up to about 100°C. The clay sample was gradually heated up in the furnace by turning on the electric current. The relative temperature and pressure readings were shown in the following data which were recorded from the pyrometer and manometer nearly every two minutes during the run.

Millivolts	Corrected Millivolts	Temp.C. Room Temp.	Hg Ht. in H. col.	Hg Ht.in L. Col.	Diff.in level of Hg Cols.	Bar. P.-diff. in level.
		20°C	84.7	9.8	74.9	0
15	27	43	84.6	9.9	74.7	2
42	54	75	84.3	10.2	74.1	8
100	112	165	83.9	11.1	72.8	21
131	143	205	83.6	11.5	72.1	28
157	169	235	83.4	11.8	71.6	33
182	194	260	83.2	12.0	71.2	37
199	211	275	83.1	12.1	71.0	39
210	222	288	83.0	12.2	70.8	41
220	232	300	82.9	12.3	70.6	43
230	242	310	82.8	12.4	70.4	45
240	252	320	82.7	12.5	70.2	47
260	272	340	82.5	12.7	69.8	51
268	280	350	82.4	12.9	69.5	54
285	297	368	82.2	13.2	69.0	59
300	312	380	82.0	13.4	68.6	63
310	322	395	81.8	13.6	68.2	67
315	327	400	81.6	13.8	67.8	71
320	332	405	81.5	13.9	67.6	73
322	334	408	81.4	14.0	67.4	75
326	338	410	81.3	14.1	67.2	77
330	342	415	81.2	14.2	67.0	79
340	352	425	81.1	14.3	66.8	81
345	357	430	80.8	14.7	66.1	88
345	357	430	80.7	14.8	65.9	90
350	362	435	80.7	14.8	65.9	90
350	362	435	80.7	14.9	65.8	91

Millivolts	Corrected Millivolts	Temp.C.	Hg Ht. in H. Col.	Hg Ht. in L. Col.	Diff. in level of Hg cols.	Bar. P.- diff. in level.
352	364	438	807	149	149	91
352	364	438	808	148	660	89
358	370	445	807	149	658	91
368	380	460	802	154	648	101
378	390	465	792	160	632	117
390	402	477	788	163	625	124
390	402	477	785	166	619	130
394	406	483	783	168	615	134
394	406	483	781	169	612	137
400	412	490	779	171	608	141
400	412	490	778	172	606	143
400	412	490	779	170	609	140
405	417	493	780	168	612	137
405	417	493	779	169	610	139
405	417	493	782	167	615	134
405	417	493	783	166	617	132
420	432	510	778	172	606	143
425	437	513	775	175	600	149
425	437	513	774	176	598	151
429	441	517	773	177	596	153
430	442	519	772	178	594	155
431	443	520	771	179	592	157

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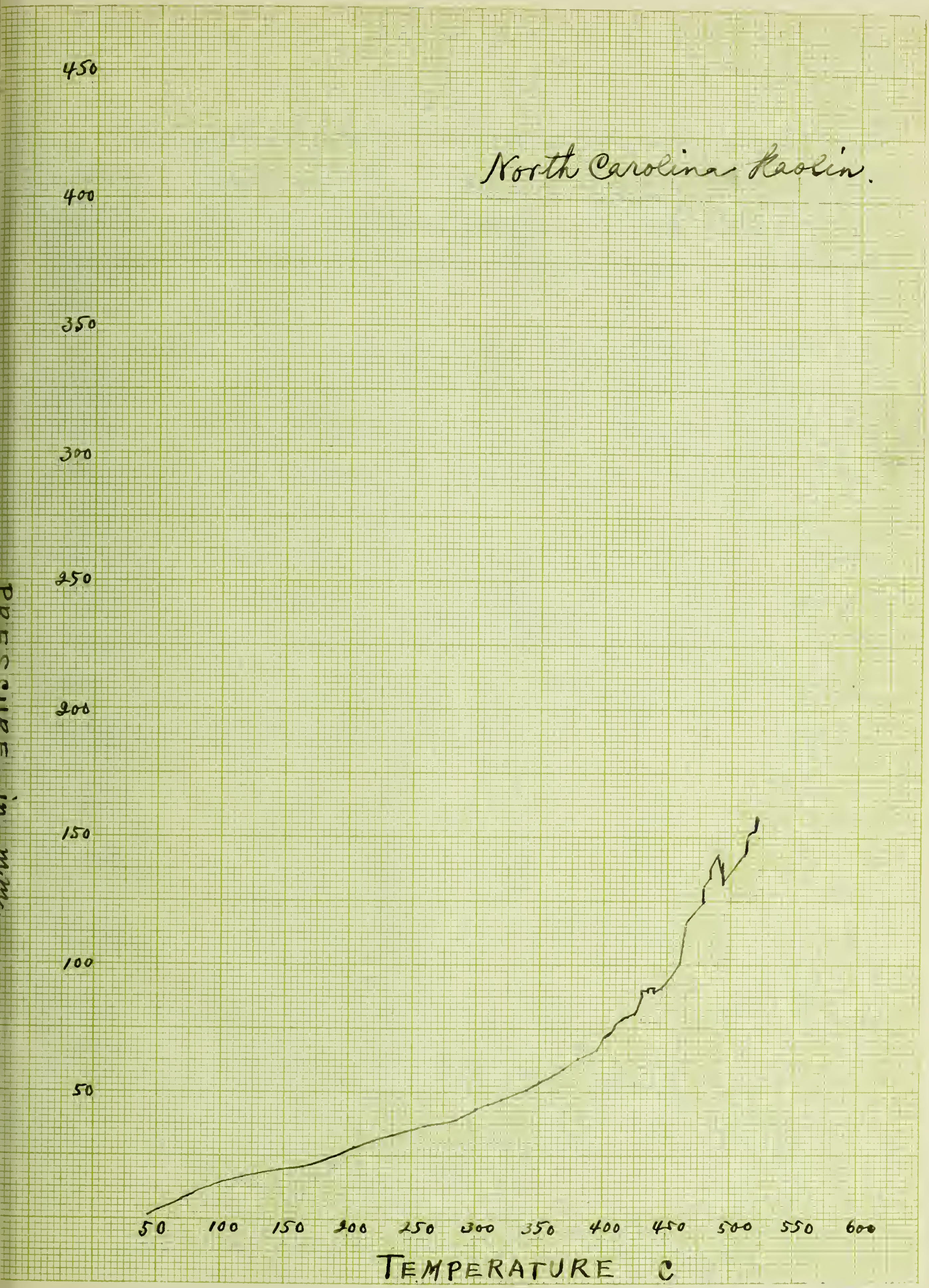
No 7


North Carolina Kaolin.
450
400
350
300
250
200
150
100
50
PRESSURE in mm.
50
100
150
200
250
300
350
400
450
500
550
600
TEMPERATURE C

Conclusion.

There are a few breaks occurring in the curve. The manometer tube was carefully watched during the determination, and at these points there was an increase in pressure followed by a decrease, the temperature remaining constant, undoubtedly due to cooling of the connecting tubes.

The first part of the curve No. 7 goes up quite smoothly, showing the gradual expulsion of the mechanically held hydroscopic moisture with the increasing temperature. From about 400° there is a rapid increase in pressure, indicating that the water vapor is given off more rapidly at this period. It is difficult to tell just when dehydration begins but the change in the rate of rise of pressure at about 400° may be significant. The run was continued until the temperature rose to 520°C. Beyond this point the furnace made of Pyrex glass would not withstand the higher temperature, so it is restricted to heat it up any more.

CHAPTER IX.

DETERMINATION OF DEHYDRATION TEMPERATURE RANGE OF HALLOYSITE.

This sample was supposed to be halloysite. Halloysite has the chemical composition $Al_2O_3.2SiO_2.XH_2O$. In this experiment the ignition loss was first determined in the following way- The halloysite given was pulverized into fine powder and two samples were weighed out. The sample was then heated in a porcelain crucible to redness for about two hours and cooled in a desiccator about half an hour. It was taken out and weighed again on a delicate balance. The results were given in percentage in terms of original weight and were stated as follows:

I. Wt. of crucible + substance to be determined = 9.9030
wt. of empty crucible = 8.4569
wt. of substance taken = 1.4461

Wt. of crucible + substance before heating = 9.9030
" " " + " after ignited = 9.7533
Loss on ignition .1497

1.4461 : .1497 = 100 : X

$$X = \frac{14.97}{1.4461} = 10.35\%$$

II. Wt. of crucible + substance to be determined = 9.4638
" " empty crucible = 8.4567
" " substance taken = 1.0071

Wt. of crucible + substance before heating = 9.4638
" " " + " after ignition = 9.3595
Loss on ignition = .1043

$$1.0071 : .1043 = 100 : X$$

$$X = \frac{10.43}{1.0071} = 10.35\ \%$$

For the determination of the dehydration temperature the whole apparatus was reassembled. A mixture of one gram of halloysite heated to 110°C and four grams of the same previously dehydrated was prepared.

This prepared sample was then introduced into the furnace. When everything was ready, the vacuum pump was started and any leakage present in the whole system was eliminated. As soon as the apparatus was evacuated, the vacuum pump was shut off and an electric current was turned on to heat the furnace. At the same time the manometer box was heated to about 100°C.

The vapor pressure produced and the corresponding temperatures were read from manometer and pyrometer, simultaneously as before and the results obtained were stated in the following data.

Millivolts	Corrected Millivolts	Temp.C. Room temp.	Hg ht.in H. Col.	Hg ht.in L. Col.	Diff.in level of Hg Cols.	Bar. P.-diff. in level
		22°C	84.2	10.5	73.7	0
35	47	70	834	114	720	17
62	74	105	815	138	677	60
82	94	135	810	145	665	72
110	122	180	810	145	665	72
180	192	260	810	145	665	72
213	225	290	810	145	665	72
225	237	303	810	145	665	72
232	244	312	808	147	661	76
240	252	322	807	148	659	78
247	259	326	806	149	657	80
250	262	330	805	150	655	82
255	267	335	805	150	655	82
260	272	342	804	151	653	84
274	286	356	803	152	651	86
280.	292	362	802	153	649	88
290	302	372	800	155	645	92
300	312	384	800	155	645	92
305	317	388	800	155	645	92
310	322	395	800	155	645	92
310	322	395	799	156	643	94
315	327	399	798	158	640	97
320	332	405	798	159	639	98
322	334	407	797	160	637	100
330	342	416	797	160	637	100
330	342	416	796	161	635	102
330	342	416	795	162	633	104

Millivolts	Corrected Millivolts	Temp.C.	Hg ht.in H. Col.	Hg ht. in L.Col.	Diff.in level of Hg Cols.	Bar. P.- diff. in level.
341	353	427	792	165	627	110
359	371	446	788	169	619	118
368	380	455	787	170	617	120
375	387	463	785	172	613	124
375	387	463	784	173	611	126
377	389	465	784	173	611	126
380	392	470	784	173	611	126
395	407	485	780	177	603	134
400	412	489	776	181	595	142
405	417	494	775	182	593	144
409	421	500	774	183	591	146
428	440	515	771	186	585	152
430	442	520	769	188	581	156
433	445	522	768	189	579	158

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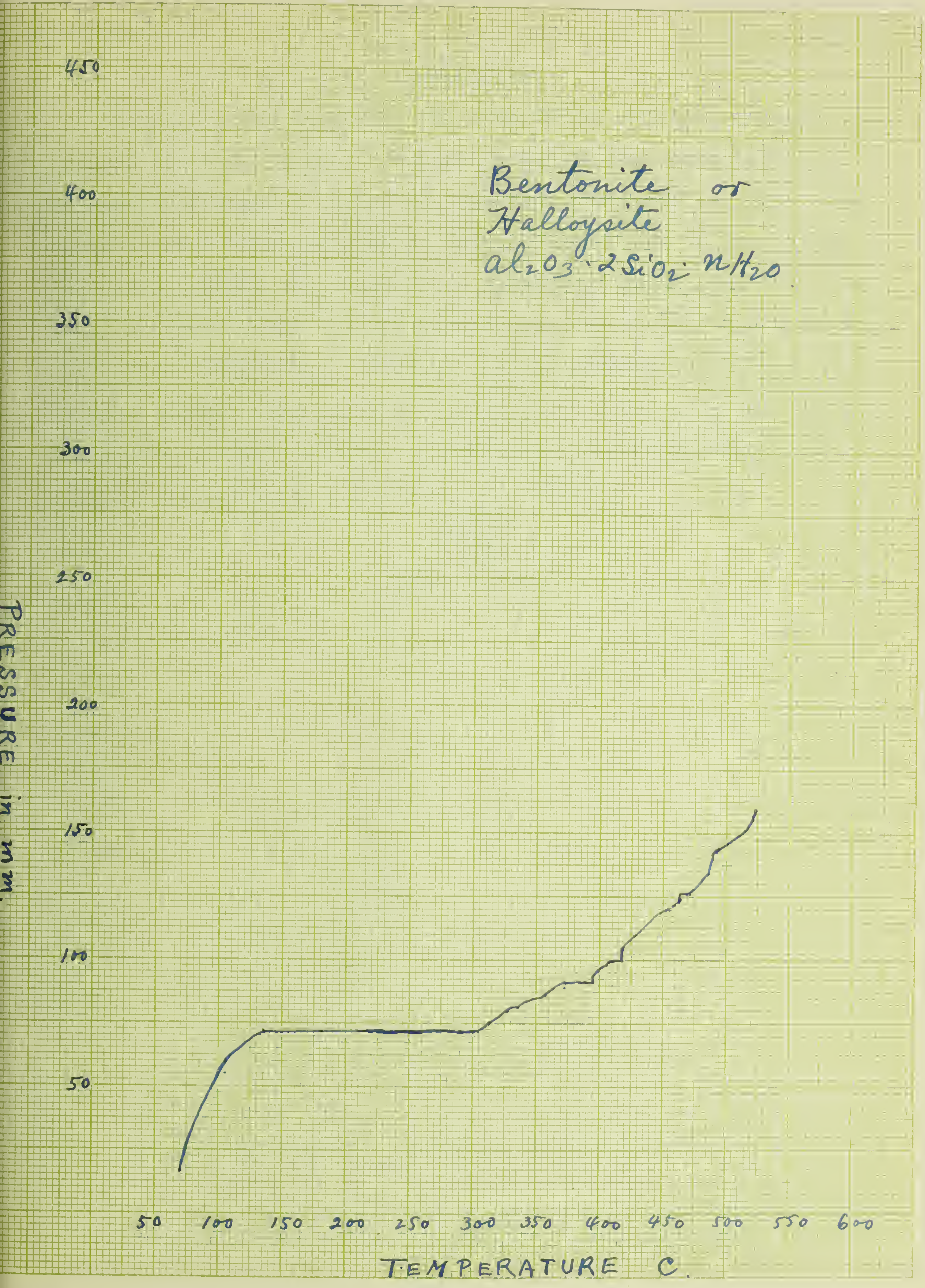
No. 8


Bentonite or
Halloysite
$al_2O_3 \cdot 2SiO_2 \cdot nH_2O$
PRESSURE in mm.
450
400
350
300
250
200
150
100
50
50
100
150
200
250
300
350
400
450
500
550
600
TEMPERATURE C.

Conclusion.

In this curve (curve No. 8) from 70° to 135° the pressure increases rapidly. This, of course is due to the rapid expulsion of hygroscopic water during that period. From 135° to 303°, the curve is flat, showing that no water is given off in that stage. From 303° upwards the curve rises gradually, indicating that more water is evolved and the pressure increases continuously with the increasing temperature, showing that dehydration is taking place in this period. The run was stopped as soon as 522°C was reached. The reason for not heating it higher than this temperature has already been given in the fore-going chapters.

CHAPTER X.

DETERMINATION OF THE DEHYDRATION TEMPERATURE RANGE OF A FLINT FIRE CLAY.

About six grams of raw flint*fire clay were taken and then ground in a mortar to fine powder. When one gram of the clay was weighed out, it was dried in an electric oven at 110°C, cooled over $CaCl_2$ in a desiccator and thoroughly mixed with four grams of dehydrated flint fire clay. The latter was done by heating it in a porcelain crucible over a blast lamp to redness for three hours.

The prepared sample was then filled into the furnace and the end of the furnace was sealed tightly. The apparatus was then ready to be reassembled and the vacuum pump was started. Until the whole apparatus was evacuated and no leaks were found the vacuum pump was shut off and the sample heated up in the furnace by turning on the electric current. At the same time the manometer box was kept at 100°C in order to prevent any condensation of water to be taking place in the apparatus.

The pressure readings on manometer and the temperature readings on pyrometer were noted respectively at the same time at intervals of about a minute. The results obtained were indicated in the following data and the accompanying diagram.

* From LaClede Christy Clay Co.

[illegible]

[illegible]

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Millivolts	Corrected Millivolts	Temp.C. Room Temp.	Hg ht.in H. col.	Hg ht.in L. column	Diff.in level	Bar. P.- diff. in level.
		22°C	847	100	747	0
.60	72	100	843	104	739	8
130	142	205	842	106	736	11
150	162	230	841	108	733	14
188	200	265	840	109	731	16
211	223	288	840	111	729	18
220	232	298	839	112	727	20
230	242	310	839	113	721	21
235	247	314	839	114	725	22
240	252	322	838	114	724	23
243	255	324	837	115	722	25
245	257	325	837	115	722	25
250	262	330	837	116	721	26
250	262	330	835	118	717	30
255	267	335	835	119	716	31
269	281	350	834	119	715	32
285	297	366	834	120	714	33
289	301	374	833	120	713	34
292	304	375	833	121	712	35
296	308	378	832	122	710	37
300	312	382	832	123	709	38
305	317	388	832	124	708	39
318	330	402	831	124	707	40
320	332	405	831	125	706	41
328	340	414	828	126	702	45
328	340	414	828	127	701	46
328	340	414	827	127	700	47

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Millivolts	Corrected Millivolts	Temp.C.	Hg Ht.in H. Col.	Hg Ht,in L. Col.	Diff. in level.	Bar. P.-diff. in level.
350	362	437	822	132	690	57
360	372	447	820	135	685	62
362	374	449	818	136	682	65
366	378	452	817	137	680	67
370	382	458	817	138	679	68
370	382	458	815	139	676	71
370	382	458	815	140	675	72
370	382	458	814	140	674	73
370	382	458	814	141	673	74
370	382	458	813	141	672	75
370	382	458	813	142	671	76
370	382	458	812	142	670	77
370	382	458	811	143	668	79
375	387	463	811	144	667	80
375	387	463	810	145	665	82
382	394	470	809	147	662	85
390	402	477	806	150	656	91
395	407	483	804	151	653	94
395	407	483	803	151	652	95
395	407	483	802	153	649	98
395	407	483	801	153	648	99
396	408	484	801	154	647	100
396	408	484	800	154	646	101
396	408	484	800	155	645	102
396	408	484	795	156	640	107
399	411	488	796	157	639	108

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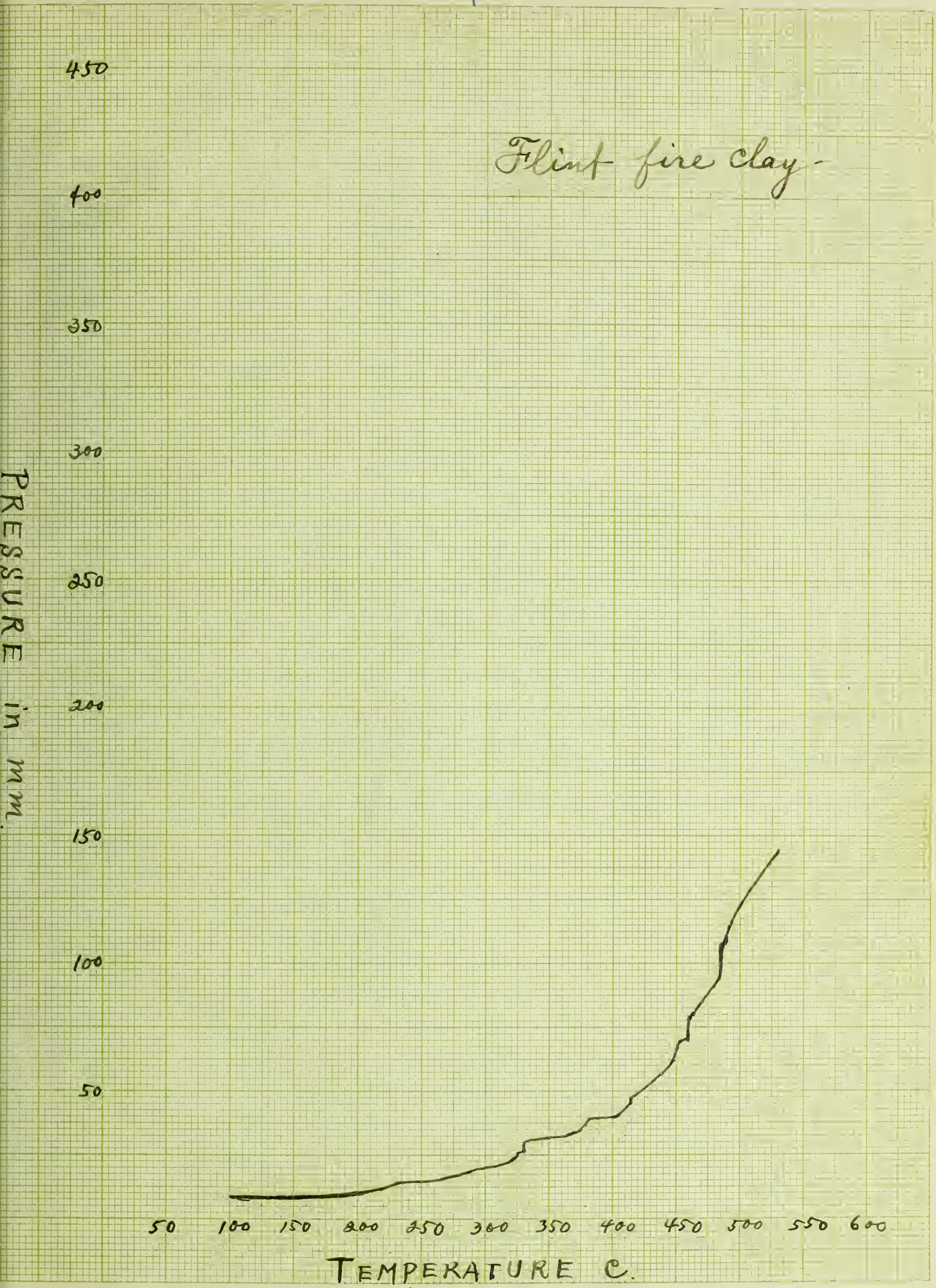
No. 9


Flint fire clay-
PRESSURE in mm.
450
400
350
300
250
200
150
100
50
50
100
150
200
250
300
350
400
450
500
550
600
TEMPERATURE C.

Millivolts	Corrected Millivolts	Temp.C.	Hg Ht.in H. Col.	Hg Ht.in L. Col.	Diff.in level.	Bar. P.-diff. in level.
400	412	489	796	157	639	108
400	412	489	795	158	637	110
400	412	489	795	159	636	111
404	416	492	791	160	631	116
436	448	524	780	171	609	138
440	452	530	777	174	603	144

Conclusion.

In this curve, we can see the pressure from 100° to 330° increases pretty slowly, showing that only a small amount of physically combined water is contained in the clay. From 330° to 400°, the curve shows that there is not much change taking place. From about 400° upwards the pressure increases more rapidly than in the former stage. This fact indicates more water is given off during this period. The dehydration of the clay took place in this temperature range. The run was stopped when the temperature reached to 530°C.

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CHAPTER XI.

RESULTS WITH APPARTUS RUN EMPTY.

The apparatus used in the determination of the dehydration point of clays was to be run without any sample in it at this time. The whole apparatus was reassembled and all connections were then firmly and securely sealed with beeswax, and resin, in order to make the apparatus air-tight.

The vacuum pump was started and let it run until the difference of the heights of the mercury in the two arms of the manometer tube was equal to the height of the mercury in a barometer. After evacuation was complete, the stop-cock D and E (Fig. 1, 6) were closed and the vacuum pump was shut off, thus sealing the vacuum in both inner and outer system.

The electric current was then turned on and the empty furnace was heated up gradually as usual. The changes in temperature and pressure were read from the pyrometer and manometer respectively at the same time and noted. The results obtained were stated in the following data and the corresponding curve was plotted in the accompanying diagram.

Millivolts	Corrected Millivolts	Temp.C. Room Temp.	Hg ht.in H.Col.	Hg.height in Low col.	Diff.in level	Bar. P.-diff.in level.
		27.5°	84 5	10 0	74 5	0
30	42	60	851	101	750	-5
45	57	85	852	102	750	-5
60	72	105	852	102	750	-5
100	112	165	852	102	750	-5
150	162	230	852	102	750	-5
190	202	270	852	102	750	-5
230	242	310	852	102	750	-5

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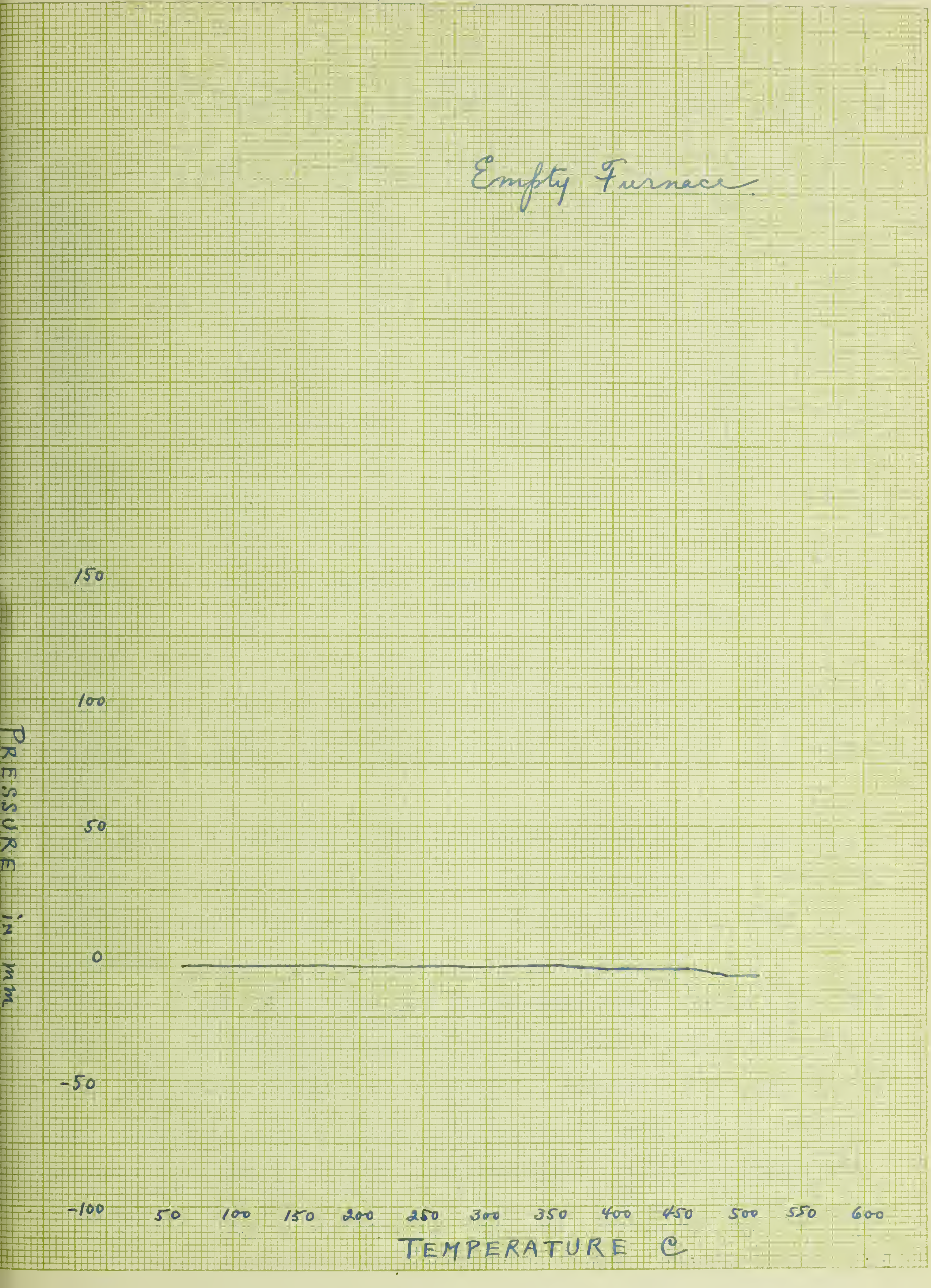
No. 10.


Empty Furnace
150
100
50
0
-50
-100
50
100
150
200
250
300
350
400
450
500
550
600
PRESSURE in mm
TEMPERATURE C

Millivolts	Corrected Millivolts	Temp.C.	Hg ht.in high Col.	Hg ht.in Low col.	Diff.in level	Bar. P.-diff. in level.
245	257	325	852	102	750	-5
260	272	342	852	102	750	-5
273	285	355	853	103	750	-5
290	302	375	854	103	751	-6
310	322	395	855	103	752	-7
319	331	404	855	103	752	-7
336	348	420	855	103	752	-7
350	362	438	855	103	752	-7
370	382	458	855	103	752	-7
390	402	478	856	103	753	-8
400	412	489	856	102	754	-9
432	444	515	856	102	754	-9

Conclusion.

In this case the curve plotted is nearly flat, because the furnace was empty and evacuated so that not any water vapor pressure was produced.

When the temperature was raised the volume of the furnace expanded gradually. The latter fact caused the mercury in the high column of manometer tube to go up and gave a negative value of pressure.

Of course, the higher the temperature rose, the more the furnace expanded, and consequently the lower the pressure would be. The curve drawn explains it very well. But since the expansion of the furnace is comparatively little, so the reduction of pressure is also very small. The run was continued until the temperature reached 515°C.

CHAPTER XII.

DETERMINATION OF THE VOLUME OF THE FURNACE.

In order to determine the volume of the furnace, a glass bulb of known volume with a three-way stop-cock was used to insert at the joint O (Fig. 6). The volume of the glass bulb was determined as follows: First weighed the empty glass bulb (Fig. 7) accurately on a delicate balance, then filled it with distilled water and weighed it again. The difference of these two weights gives the weight of distilled water.

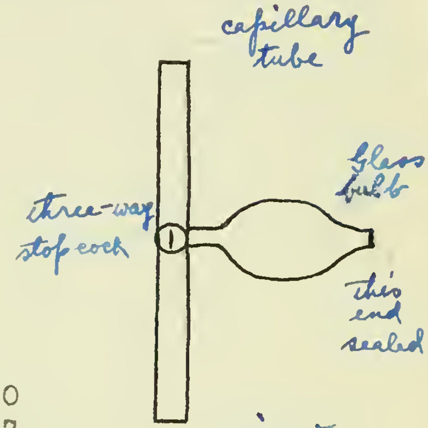


Fig 7

Room Temp. = 21°C.

Wt. of bulb + water = 154.7730
" " empty bulb = 62.4467
" " water = 92.3263 grams

Since one gram of water at 4°C occupies 1c.c. and one gram of water at 21°C occupies 1.0019c.c; 92.3263 g. of water at 21° occupy 92.3263 X 1.001981 = 92.509c.c.

After the water in glass bulb was poured out and the latter was dried, the bulb containing air was inserted at the place mentioned above and the connection between the capillery tube and the glass bulb was cut off by closing the three-way stop-cock.

As soon as the whole apparatus was assembled, it was evacuated except the glass bulb by starting the vacuum pump. After the evacuation was complete, the manometer gave the same reading as that of the barometer, that 845 - 100 = 745 mm.

Then the stop-cocks D and E were closed (Fig. 1, or 6) and the vacuum pump was shut off. Then the three-way stop-cock was opened by tunning it over 180° and the air contained in the glass

[illegible]

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bulb would occupy the whole furnace. This latter fact naturally pressed down the high Hg columns and caused the manometer reading to drop, that is, it gave 614 - 320 = 294 mm.

Now let V_1 = the volume of bulb

V_2 = the volume of bulb + volume of furnace

V_3 = the volume of furnace.

By formula $$\frac{V_1}{V_2} = \frac{P_2}{P_1}$$

$$\frac{92.5}{V_2} = \frac{294}{745}$$

Therefore $V_2 = \frac{92.5 \times 745}{294} = 234.3$ c.c.

Since $V_3 = V_2 - V_1$

Therefore $V_3 = 234.3 - 92.5 = 141.8$ c.c.

CHAPTER XIII.

CALULATION OF THE AMOUNT OF WATER VAPOR SET FREE AT VARIOUS TEMPERATURES.

With the data available for the various clays, the amount of water vapor set free at various temperatures can be calculated. In the case of Tennessee ball clay No. 3, I am unable to calculate the amount of water given off at different temperatures, because the furnace broke so that its volume could not be determined. The calculation for other four clays are stated in the following and the graphic representation of the relative amount of water evolved at different temperatures for various clays is shown in the curves plotted (No. 11) by using temperature VS mole of water. In the calculation, I have not made any reduction of the volume of the solid material from the volume of the furnace, because the true volume of the material is comparatively so small that it will not make an appreciable difference in the result.

Calculation:

(1) Georgia Kaolin

Let n = no. of mols of water vapor set free at 145°C and 32 mm., $\pi \times \overline{.15}^2 \times 1.4$ = volume of a part of manometer occupied by the vapor.

$PV = n\ R\ T.$

$n = \frac{PV}{RT}$

Here P = 3.2 cm., $V = 141.8 + \pi \times \overline{.15}^2 \times 1.4 = 141.8 + .1 = 141.9$ c.c. R = 84720 T = 273 + 145 = 418°.

$$n = \frac{3.2 \times 13.6 \times 141.9}{84720 \times 418} = \frac{6175.488}{35412960} = .000179 \text{ mols.}$$

At 305°C 41 mm.

$n = \frac{PV}{RT}$ Here P = 4.1 cm.
R = 84720

$V = 141.8 + \pi \times .\overline{15}^2 \times 1.8 = 141.8 + .1 = 141.9$ cc.

$T = 273 + 305 = 578.$

$$\therefore n = \frac{4.1 \times 13.6 \times 141.9}{84720 \times 578} = \frac{7912.344}{48968160} = .000163 \text{ mols.}$$

at 404°C. 73 mm.

$n = \frac{PV}{RT}$ Here $P = 7.3$ cm $V = 141.8 + \pi \times .\overline{15}^2 \times 3.3 = 141.8 + .2 = 142$ c.c.

$R = 84720$ $T = 273 + 404 = 677$

$$\therefore n = \frac{7.3 \times 13.6 \times 142}{84720 \times 677} = \frac{14097.76}{57355440} = .000245 \text{ mols.}$$

at 453°C 140 mm.

$n = \frac{PV}{RT}$ Here $P = 14.0$ cm $V = 141.8 + \pi \times .\overline{15}^2 \times 6.8 = 141.8 + .5 = 142.3$ c.c.

$R = 84720$ $T = 273 + 453 = 726$

$$\therefore n = \frac{14 \times 13.6 \times 142.3}{84720 \times 726} = \frac{27093.92}{61506720} = .000440 \text{ mols.}$$

at = 508°C 240 mm.

$n = \frac{PV}{RT}$ Here $P = 240$ mm $V = 141.8 + \pi \times .\overline{15}^2 \times 12.0 = 141.8 + .9 = 142.7$ c.c.

$R = 84720$ $T = 273 + 508 = 781$

$$\therefore n = \frac{24 \times 13.6 \times 142.7}{84720 \times 781} = \frac{46577.28}{66166320} = .000703 \text{ mols.}$$

(2) North Carolina Kaolin.

at 165°C 21 mm.

$n = \frac{PV}{RT}$ Here $P = 2.1$ cm $V = 141.8 + \pi \times .\overline{15}^2 \times .6 = 141.8 + .04 = 141.84$ c.c.

$R = 84720$ $T = 273 + 165 = 438$

$$\therefore\ n = \frac{2.1 \times 13.6 \times 141.84}{84720 \times 438} = \frac{4050.9504}{37107360} = .000109 \text{ mols.}$$

at 300°C 43 mm.

$n = \frac{PV}{RT}$ Here $P = 4.3$ cm $V = 141.8 + \pi \times .\overline{15}^2 \times 1.6 = 141.8 + .1 = 141.9$ c.c.

$R = 84720$ $T = 273 + 300 = 573$

$$= \frac{4.3 \times 13.6 \times 141.9}{84720 \times 573} = \frac{8298.312}{48544560} = .000170 \text{ mols.}$$

at 405°C 73 mm.

$n = \frac{PV}{RT}$ Here $P = 7.3$ cm $V = 141.8 + \pi \times .\overline{15}^2 \times 3.0 = 141.8 + .2 = 142$ c.c.

$R = 84720$ $T = 273 + 405 = 678$

$$\therefore\ n = \frac{7.3 \times 13.6 \times 142}{84720 \times 678} = \frac{14097.76}{57440166} = .000245 \text{ mols.}$$

at 460°C. 101 mm.

$n = \frac{PV}{RT}$ $P = 10.1$ $V = 141.8 + \pi \times .\overline{15}^2 \times 4.3 = 142.1$ c.c.

$R = 84720$ $T = 273 + 460 = 733$

$$\therefore\ n = \frac{10.1 \times 13.6 \times 142.1}{84720 \times 733} = \frac{19518.856}{62099760} = .000314 \text{ mols.}$$

at 510°C 143 mm.

$n = \frac{PV}{RT}$ Here $P = 14.3$ $V = 141.8 + \pi \times .\overline{15}^2 \times 617 = 141.8 + .5 = 142.3$ c.c.

$R = 84720$ $T = 273 + 510 = 783$

$$\therefore\ n = \frac{14.3 \times 13.6 \times 142.3}{84720 \times 783} = \frac{27674.504}{66335760} = .00417 \text{ mols.}$$

(3) Halloysite.

at 135° 72 mm.

$n = \frac{PV}{RT}$ Here $P = 7.2$ cm $V = 141.8 + \pi X \overline{.15}^2 X 3.5 = 141.8 + .2 = 142$ c.c.

$R = 84720$ $T = 273 + 135 = 408$

$\therefore n = \frac{7.2 X 13.6 X 142}{84720 X 408} = \frac{13904.64}{33565760} = .000415$ mols.

at 303° 72 mm.

$n = \frac{PV}{RT}$ Here $P = 7.2$ cm $V = 141.8 + \pi X.\overline{15}^2 X 3.5 = 141.8 + .2 = 142$ c.c.

$R = 84720$ $T = 273 + 303 = 576$

$\therefore n = \frac{7.2 X 13.6 X 142}{84720 X 576} = \frac{13904.64}{48798720} = .000285$ mols.

at 399° 97 mm.

$n = \frac{PV}{RT}$ Here $P = 9.7$ cm $V = 141.8 + \pi X.\overline{15}^2 X 4.7 = 141.8 + .3 = 142.1$ c.c.

$R = 84720$ $T = 273 + 399 = 672$

$\therefore n = \frac{9.7 X 13.6 X 142.1}{84720 X 672} = \frac{18745.832}{56931840} = .000329$ mols.

at 446°C 118 mm.

$n = \frac{PV}{RT}$ Here $P = 11.8$ cm $V = 141.8 + \pi X.\overline{15}^2 X 5.7 = 141.8 + .4 = 142.2$ c.c.

$R = 84720$ $T = 273 + 446 = 719$

$\therefore n = \frac{11.8 X 13.6 X 142.2}{84720 X 719} = \frac{23202040}{60913680} = .000380$ mols.

at 500°C 146 mm.

$n = \frac{PV}{RT}$ Here $P = 14.6$ $V = 141.8 + \pi X.\overline{15}^2 X 7.1 = 141.8 + .5 = 142.3$ c.c.

$R = 84720$ $T = 273 + 500 = 773$

$\therefore n = \frac{14.6 \times 13.6 \times 142.3}{84720 \times 773} = \frac{28255.088}{65488560} = .000431$ mols.

(4) Flint fire clay.

at 205°C 11mm.

$n = \frac{PV}{RT}$ Here $P = 1.1$ cm $V = 141.8 + \pi \times .15^2 \times .3 = 141.8 + .02$
$= 141.82$ c.c.

$R = 84720$ $T = 273 + 205 = 478$

$\therefore n = \frac{1.1 \times 13.6 \times 141.82}{84720 \times 478} = \frac{2121.7372}{40496160} = .000052$ mols.

at 298°C 20mm.

$n = \frac{PV}{RT}$ Here $P = 2.0$ cm. $V = 141.8 + \pi \times .15^2 \times .6 = 141.8 + .04$
$= 141.84$ c.c.

$R = 84720$ $T = 273 + 298 = 571$

$\therefore n = \frac{2 \times 13.6 \times 141.84}{84720 \times 571} = \frac{3858.048}{48375120} = .000079$ mols.

at 402°C 40mm.

$n = \frac{PV}{RT}$ Here $P = 4.0$ cm $V = 141.8 + \pi \times .15^2 \times 1.4 = 141.8 + .1$
$= 141.9$ c.c.

$R = 84720$ $T = 273 + 402 = 675.$

$\therefore n = \frac{4 \times 13.6 \times 141.9}{84720 \times 675} = \frac{7719.36}{57186000} = .000134$ mols.

at 449°C 65mm.

$n = \frac{PV}{RT}$ Here $P = 6.5$ cm $V = 141.8 + \pi \times .15^2 \times 2.7 = 141.8 + .2$
$= 142$ c.c.

$R = 84720$ $T = 273 + 449 = 722$

$\therefore n = \frac{6.5 \times 13.6 \times 142}{84720 \times 722} = \frac{12552.80}{61167840} = .000205$ mols

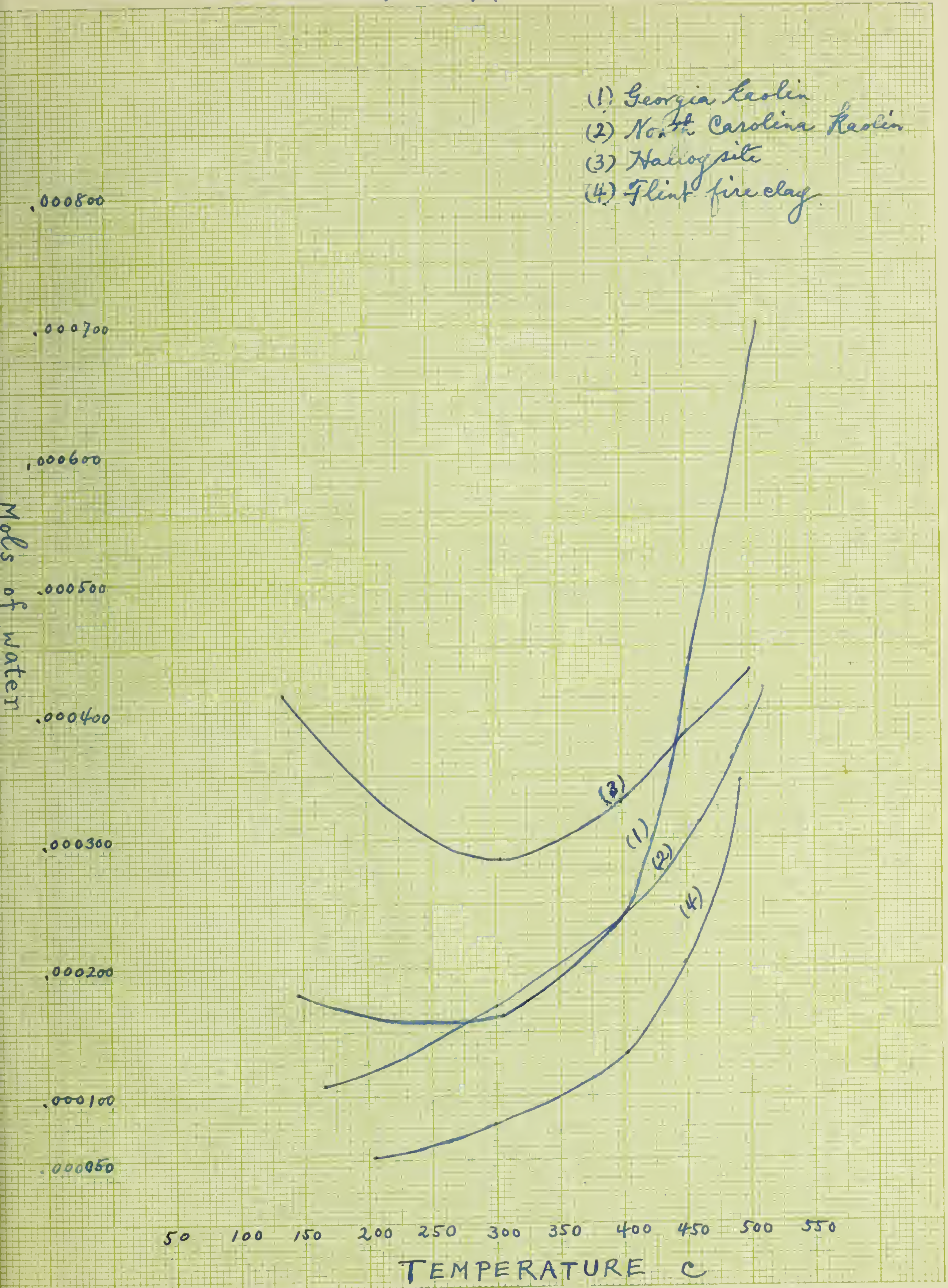
at 492°C   116 mm.

$n = \frac{PV}{RT}$ Here $P = 11.6$ cm $V = 141.8 + \pi X .15^2 X 5.4 = 141.8 + .4$
$= 142.2$ c.c.

$R = 84720$ $T = 273 + 492 = 765$

$$= \frac{11.6 X 13.6 X 142.2}{84720 X 765} = \frac{22433.472}{64810800} = .000346 \text{ mols}$$

No 11.


(1) Georgia kaolin
(2) North Carolina kaolin
(3) Halloysite
(4) Flint fire clay
.000800
.000700
.000600
.000500
.000400
.000300
.000200
.000100
.000050
Mols of water
(1)
(2)
(3)
(4)
50
100
150
200
250
300
350
400
450
500
550
TEMPERATURE C

CHAPTER XIV.

SUMMARY.

Not all of the water present in clay is a part of the clay base in the sense of water of hydration, since one part of the water contained in the clay is only hygroscopic water. Like other hydrated compounds, clay does not possess a definite temperature at which the dehydration will take place and will be complete, but dehydration will depend upon the conditions of temperature and pressure to which it is subjected.

The diagrams bring out this fact clearly, that the combined water of the clay substance is not expelled at a definite temperature but its loss depends upon equilibrium conditions governed by the pressure as well as temperature.

The first part in the curves represents the pressure produced by physically held water and the remaining part of the curve represents that produced by chemically combined water. The small breaks in the curves indicate a decrease in pressure. This is caused undoubtedly by variations in heating and cooling of the system. The pressures shown on the

dehydration curves of different kinds of clays at corresponding temperatures could not be compared each to each exactly, because the sample of clay taken in each case varies slightly about one tenth of a gram. The temperature at which dehydration begins with the different clays, however, can be seen clearly in some cases in the separate curves.

So far the dehydration curves of five different kinds of clays have been determined, namely, Tennessee ball clay No. 3, Georgia kaolin, North Carolina kaolin, halloysite and flint fire clay from Missouri. Among the various clays, it is apparant that the flint fire clay possess similar dehydration temperature range as that of kaolin (curve No.11), although the number of moles of water set-free and the pressure produced by it is different. In the case of halloysite it behaves differently.

The exceedingly small amount of water given off (Chapter XIII) in heating the clays at various temperatures shows that dehydration is quite incomplete. It seems to be due to the fact that there is an equilibrium condition existing in the system which consists of three components-namely, water vapor, hydrated and dehydrated materials. By applying the formula

$$\frac{d \log_e K_p}{dT} = \frac{HP}{RT^2}$$ which integrated gives

$$2 \log p = \frac{HP}{2R} \frac{1}{T} \qquad \frac{HP}{2R} = m$$ (a constant)

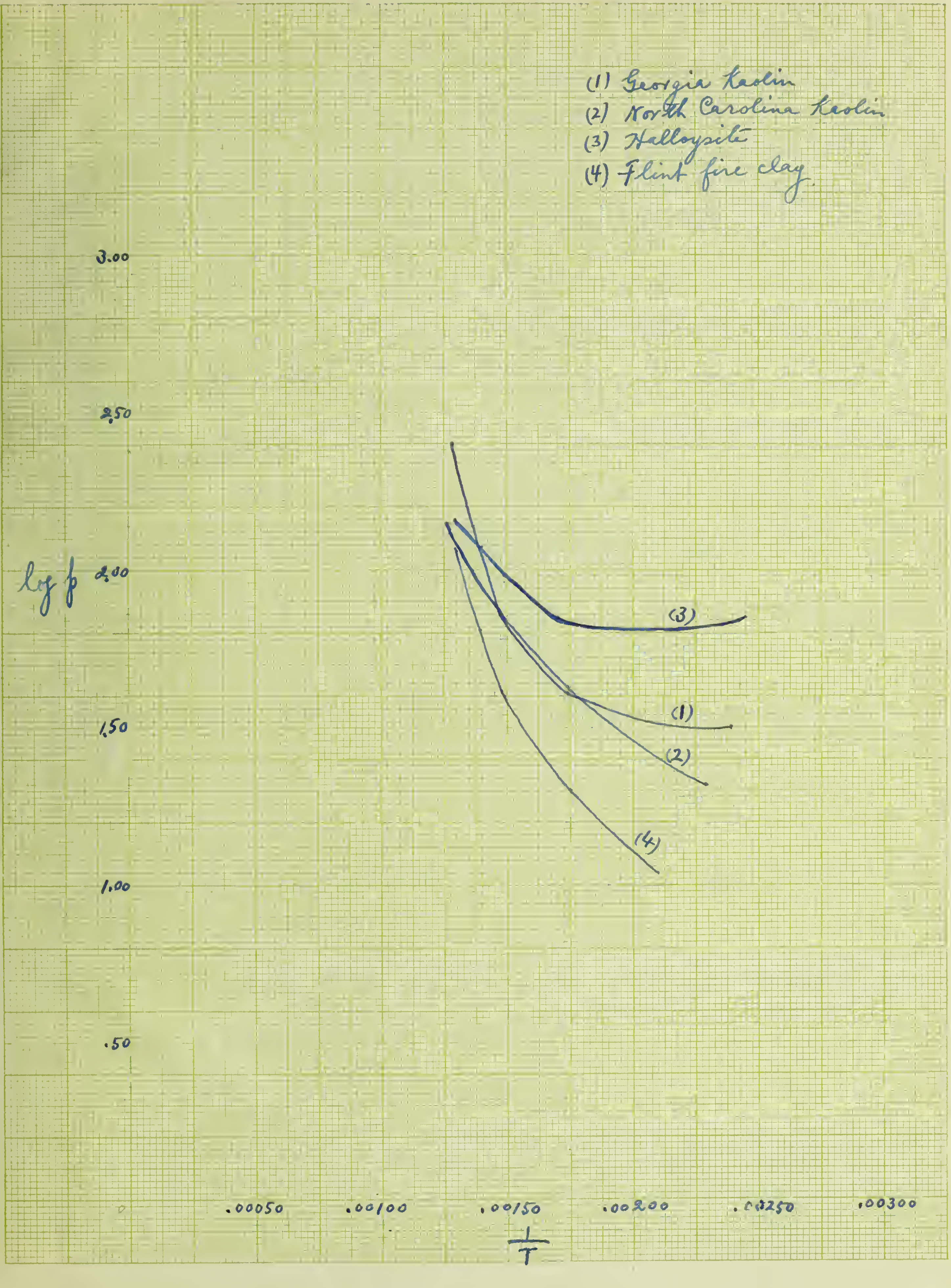
$Y = mx$ (the equation of a straight line)

we may calculate the value of log p and $\frac{1}{T}$ at different temperatures and plot the results. We find that the graph (curve No. 12) for various clays shows straight line above 300°C with curve (2) and

(3), and above 400° with curve (1) and (4), thus confirming the view just stated above. The portion of the curve below 300°C represents loss of hygroscopic moisture.

J. W. Mellor (Eng. Trans. Cer. Soc. Vol. 16, p.73) attempts to classify clayey materials according to the results of thermal analysis. The results here obtained do not show the differences in character of various types of clays which might reasonably be expected from Mellor's observation.

No. 12.


(1) Georgia Kaolin
(2) North Carolina Kaolin
(3) Halloysite
(4) Flint fire clay
3.00
2.50
log p
2.00
1.50
1.00
.50
0
.00050
.00100
.00150
.00200
.00250
.00300
$\frac{1}{T}$
(3)
(1)
(2)
(4)

(1) Georgia Kaolin

at 145°C 32 mm.

log 32 = 1.5051

$$\frac{1}{T} = \frac{1}{273 + 145} = \frac{1}{418} = .00238$$

at 305°C. 41 mm

log 41 = 1.6128

$$\frac{1}{T} = \frac{1}{273 + 305} = \frac{1}{578} = .00173$$

at 404°C. 73 mm.

log 73 = 1.8633

$$\frac{1}{T} = \frac{1}{273 + 404} = \frac{1}{677} = .00147$$

at 453°C 140 mm.

log 140 = 2.1461

$$\frac{1}{T} = \frac{1}{273 + 453} = \frac{1}{726} = .00137$$

at 508°C 240 mm.

log 240 = 2.3802

$$\frac{1}{T} = \frac{1}{273 + 508} = \frac{1}{781} = .00128$$

(2) North Carolina Kaolin

at 165°C 21 mm.

log 21 = 1.3222

$$\frac{1}{T} = \frac{1}{273 + 165} = \frac{1}{438} = .00228$$

at 300°C 43 mm.

log 43 = 1.6335

$$\frac{1}{T} = \frac{1}{273 + 300} = \frac{1}{573} = .00174$$

at 405°C 73 mm.

log 73 = 1.8633

$$\frac{1}{T} = \frac{1}{273 + 405} = \frac{1}{678} = .00147$$

at 460° 101 mm.

log 101 = 2.0043

$$\frac{1}{T} = \frac{1}{273 + 460} = \frac{1}{733} = .00136$$

at 510°C 143 mm.

log 143 = 2.1553

$$\frac{1}{T} = \frac{1}{273 + 510} = \frac{1}{783} = .00127$$

(3) Halloysite

at 135°C 72 mm.

log 72 = 1.8573

$$\frac{1}{T} = \frac{1}{273 + 135} = \frac{1}{408} = .00245$$

at 303°C 72 mm

log 72 = 1.8573

$$\frac{1}{T} = \frac{1}{273 + 303} = \frac{1}{576} = .00173$$

at 399°C 97 mm.

log 97 = 1.9868

$$\frac{1}{T} = \frac{1}{273 + 399} = \frac{1}{672} = .00148$$

at 446°C 118 mm.

log 118 = 2.0719

$$\frac{1}{T} = \frac{1}{273 + 446} = \frac{1}{719} = .00139$$

at 500°C 146 mm.

log 146 = 2.1644

$$\frac{1}{T} = \frac{1}{273 + 500} = \frac{1}{773} = .00129$$

(4) Flint fire clay.

at 205°C 11 mm.

log 11 = 1.0414

$$\frac{1}{T} = \frac{1}{273 + 205} = \frac{1}{478} = .00209$$

at 298°C 20 mm.

log 20 = 1.3010

$$\frac{1}{T} = \frac{1}{273 + 298} = \frac{1}{571} = .00175$$

at 402°C 40 mm.

log 40 = 1.6021

$$\frac{1}{T} = \frac{1}{273 + 402} = \frac{1}{675} = .00148$$

at 449°C 65 mm.

log 65 = 1.8129

$$\frac{1}{T} = \frac{1}{273 + 449} = \frac{1}{722} = .00138$$

at 492°C 116 mm.

log 116 = 2.0645

$$\frac{1}{T} = \frac{1}{273 + 492} = \frac{1}{765} = .00130$$